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SEPARATOR DEVELOPMENT FOR A HEAT STERILIZABLE BATTERY

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ABSTRACT

A continuous coating apparatus to prepare sterilizable battery separators has been built, tested and put into the production of several feet of separator material which exhibited satisfactory properties.

The effect of time of immersion in extractant solution upon separator composites has been investigated using formulations in which the filler was either Zeolon H or coarse Zirconia. The best extracting solutions contained dioxane. Generally no correlation was found between separator properties and time of immersion.

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I. INTRODUCTION

This Quarterly Report covers the work performed during the period July 1 to September 30, 1967. The effort expended during this time and reported here includes three main topics:

1. Collecting the necessary ingredients and organizing, setting up and improving testing and manufacturing apparatuses.
2. Investigating the effect of immersion time in a variety of extracting solutions upon separator materials made from different formulations.
3. Carrying out continuous coating runs using narrow Webril tape, with the aim of establishing criteria for the continuous production of one foot wide separator material.

II. PROGRESS

1. Materials

1.1 Support

On the basis of previous findings it was decided to make separator materials consisting of a supporting tape coated by a polymer: inorganic filler: solvent mixture. The chosen tape was Webril (Kendall Fiber Product Division), which is a non-woven polypropylene fabric with

a nominal thickness of 1.4-1.5 mils. Two hundred yards of 12 inch wide Webril (SM1262) were acquired, and samples for coating and testing are at present obtained from the original roll by slicing it into 3 inch wide sections, used especially in the continuous runs. This material seems entirely satisfactory from the points of view of strength, porosity and coatability. Since cutting this material into narrower widths produces uneven edges, 3 inch wide Webril tape will be ordered.

The Kendall Company offers many other types of fabrics; they will be investigated if the results of the present work warrant it.

1.2 Fillers

Inorganic fillers were chosen to act both as reinforcing agents and as porosity foci; on the basis of previous work the most promising inorganic filler was hydrated zirconia supplied by the Bio Rad Company in 100-200 mesh particle size (H20-1 ion exchange crystals, \$25/lb). However this material, which was ordered last July, has not yet been received. In its place some 20-50 mesh zirconia was sent by the same company (\$12/lb), and this material, which loses 20% of its weight on heating for 20 hours in runs at 110°C, was successfully used in several formulations.

Another type of zirconia was purchased and tested, namely "special" hydrous form ZrO_2 from the Titanium Alloy Division, National Lead Company (\$3/lb). This filler contained a large amount of absorbed water; in fact 38.5% of the filler's weight was lost after heating for 18 hrs at 180°C in runs. The TAM zirconia as well was used successfully as a filler in separator coating formulations. Zeolon H pigment was also used satisfactorily in coating formulations (weight loss is 11.1% after heating at 120°C for 18 hours); however, in view of the fact that Zeolon H was found to be more soluble in concentrated caustic than hydrated zirconia, the work done using Zeolon was mainly carried out for practice and to settle some procedural points.

It must be noted that drying of any of the fillers mentioned above, and prolonged ball milling in the presence of the binder

solution, are of paramount importance if one wishes to obtain smooth coatings of reproducible quality.

1.3 Organic polymeric binders

The polymer used previously with best results as a separator binder has been a polysulfone (Pl700) supplied in pellet form by the Union Carbide Company. This material consists of aromatic rings connected to each other by ether, isopropylidene and sulfone linkages. While the sulfone and ether links are known to be resistant to thermal and oxidative attack and to the attack of concentrated alkali, the isopropylidene group constitutes a weak point in the polymer chain in this respect. Therefore we were interested in acquiring a sample of a polysulfone (polymer 380) which the 3M company has been supplying in limited quantities and which consists of the usual aromatic chain with ether, sulfone and diphenyl linkages without the relatively labile isopropylidene group. This material was supplied to us in the form of a thin film which dissolves in DMAC relatively easily and will be used as the binder in some future preparation. The 3M company also makes polymer 360 (in pellets or in powder form), which is similar to Polymer 380 but is reputed to have a lower molecular weight. This material costs about \$30/lb in developmental quantities and samples have been acquired for testing.

1.4 Formulations

All the coating formulations reported here were made using polysulfone solutions consisting of 15g of polysulfone per 100g of DMAC. The dried oxide is added to the solution in 20:15, 30:15, and 45:15 ratios (or 1:33, 2:1, and 3:1) between the filler and the solid polymer. For example, a formulation may consist of 80g of dried zirconia in 460g of polysulfone solution (containing 60g of the binder), so that the filler:binder ratio in this case is 20:15. This amount of material is generally sufficient for one continuous run of several dozen feet; however, when experimental runs are longer, the practice has been to use

fresh batches as often as possible because the solvent is very hydrophilic and exhibits a relatively high vapor pressure, thereby causing the premature precipitation or coagulation of the binder.

2. Continuous Coating Apparatus

Figure 1A shows a sketch of the continuous coating machine built during the past month. The whole set-up is mounted on a supporting frame which is fastened to a movable platform. The Webril tape 3 is wound around the payoff roller and is kept under tension by a friction brake 2. The tape then passes through an arrangement consisting of an 8 inch long Gardner film casting knife 5 and a 9 inch long aluminum angle 6 one edge of which is butted lengthwise, at an angle, against the edge of the Gardner applicator's knife. This set-up serves to adjust with great accuracy the gap through which the tape slowly travels and which limits the amount of binder:filler:solvent mix 4 to be deposited on the tape. It was ascertained from the company which manufactures the Gardner knife, that they will make to order identical knives in larger sizes; at the appropriate time a casting knife able to accommodate 12 inch wide tape will be ordered and will be incorporated in the continuous coating machine.

The coated tape then dips into tank 8, being led parallel to the bottom of the tank by two 1/2 inch Teflon guide rods 7. The tank is filled with the appropriate solvent mixture which precipitates the organic binder out of its DMAC solution and, at the same time, extracts the DMAC solvent thus causing porosity to be formed in the binder:filler layer. The position of the two rods 7 relative to each other is one of the many parameters governing the duration of the dip of the tape and, therefore, the degree of porosity of the separator.

The treated tape then leaves tank 8 and travels between two rollers 9 the gap between which can be adjusted. Thus pressure can be exerted continuously upon the composite to improve its conductivity. Finally, the treated separator tape is rolled up by a 2 inch diameter take up roller driven by a high torque, low speed motor. In the course

of some preliminary runs with this continuous coating machine it was found that the treated tape was not completely dry as it wound around the take up roller; in addition, the solvent mixture inside tank 8 became contaminated by fragments and floating debris from the coating mix. To combat these two drawbacks, heaters have been installed on either side of the tape halfway between the compression rollers 9 and the take up roller 10, and a circulating pump equipped with filter has been installed at tank 8. On a large scale operation consideration should be given to monitoring the composition of the solvent mixture in the tank so as to keep the ingredient proportions constant.

3. Effect of Extraction Time

The manner in which the coated tape acquires its porosity is envisioned as being a result of a capillary extraction of the solvent, originally present in the coating mixture, by an extractant solution. A simultaneous effect of the immersion in the extracting bath is the coagulation or precipitation of the organic binder to give a polymeric coating which appears continuous to the naked eye.

Since the porosity acquired by this method is also dependent on the type, concentration, particle size of the inorganic filler, it is conceivable that both time of immersion and type and concentration of extracting solutions have a bearing on the resistivity and ion diffusion characteristics of separator composites.

Therefore the effect of time of extraction in several types of extracting solutions, as evidenced by the variation of resistivity and ion diffusion values, was investigated.

3.1 Procedure

The coating device consisted of two stainless steel shimstock blades set edge on edge at a predetermined distance from each other and secured by screws to a common handle. The piece of Webril is inserted in the gap between the blades and is held in a vertical position;

several portions of the coating mix are poured on the blades on either side of the handheld Webril sample, which is then pulled down slowly and evenly. This action causes the Webril tape to be covered on either side by a smooth, even coat whose thickness is determined by the gap between the blades. It is of paramount importance that: (1) the coating mixture be continually agitated to prevent settling of filler (this is usually done magnetically); (2) that new coating mixtures be made frequently in order to avoid problems due to evaporation of solvent; (3) that after each coating the blades and the gap be cleaned carefully, since the polysulfone resin easily solidifies out of solution thereby causing rough coatings on the next sample.

The procedure mentioned above, laborious though it is, does supply coated samples of relative uniform thicknesses possessing good continuity. Air bubbles in the original, magnetically agitated mixture do not seem to cause trouble. The ingredient composition ratio chosen yields a coating mixture which is fluid enough so that air bubbles rise and disappear easily; its viscosity gives smooth and continuous coatings in a relatively uniform manner.

After the Webril sample is coated, it is then extracted for the required length of time in the predetermined extractant solution. The latter solutions were made to a total volume of 600 cc each and are contained in wide mouth, screw cap jars. The jars containing samples designed to be extracted for a relatively long time are shaken vigorously from time to time. At the end of the extraction time the coated samples are removed from the jars, dried gently between sheets of absorbent paper, their thickness measured in five positions, and then they are stored between paper sheets under a light weight to prevent curling.

Each extraction run is made in duplicate.

The shim stock blade gap chosen was 3 mils; previous attempts with 6 mil gap resulted in the mixture running unevenly down the Webril sample.

3.2 Extraction Schedule

The following is the schedule followed for the investigation of extraction time effect:

1. Time of extraction: 1 minute, 30, 120 and 360 minutes

2. Extraction solutions (by volume)

50:50 Water:DMAC

25:75 Water:DMAC

75:25 Water:DMAC

50:50 Water:DMSO

25:75 Water:DMSO

75:25 Water:DMSO

50:50 Water:Dioxane

25:75 Water:Dioxane

75:25 Water:Dioxane

50:50 Water:Acetic acid

25:75 Water:Acetic acid

75:25 Water:Acetic acid

Samples were coated and air-dried (instead of being extracted) to be used as standards.

3.3 Measurements

Resistivity Measurements

It was early noticed that some of the experimental composites had such a high resistance that it was not possible to measure them using the more sensitive dc or ac tests described previously. Accordingly, a simplified tester was used which indicates the resistance by the measured potential across a cell when a current flows between electrodes. The test circuit consists of a battery, a variable resistance, a voltmeter and a milliammeter. When the current is set at 10 ma, each volt on the meter corresponds to 100 ohms; this renders the estimate of composite resistance fairly quick. All the samples were presoaked in

40% KOH for 24 hours, and three positions on each sample were measured for resistance. No sample was pressed.

Electrolyte Diffusion Time

The degree or extent of diffusion of hydroxyl ions through the coated membranes gives an indication of whether there are relatively large uncoated portions on the composite, or whether the permeability of the membranes is too low. The measurement of such a parameter is, in other words, a quality control test. This test was carried out by measuring as a function of time, the increase in pH units in a cell initially full of distilled water and separated from another cell initially full of 40% KOH by an experimental separator membrane.

3.4 Results and Discussion

3.4.1 Zeolon H-containing Formulations

The formulation used was Zeolon H:PS:DMAC (20:15:100) coated on nominal 1.5 mil Webril tape.

Table 1 presents the results obtained from the extraction time experiment in the presence of twelve different extractant compositions. This table gives, in addition to resistance and specific resistivity values, the thickness in mils of each coated and extracted sample, taken in five different positions on the sample. An attempt was made to measure the thickness of the coated samples before their extraction; however, the reliability of such measurements is very low, because the pressure of the thickness tester plate is such that the still liquid coating on the sample is easily displaced. It was therefore impossible to arrive at a relationship between thicknesses before and after extraction as a function of time of extraction and type of extractant.

It will be noticed that the column under the heading "Specific Resistivity" is divided into two sections. The one on the left contains the specific resistivity values based on the average of all three resistance values listed at its left; the column on the right, on the

other hand, lists the specific resistivity values derived from the average of the last two resistance values of the corresponding sample. The reason for this division is that of the forty eight samples tested and reported here, only three have a first resistance reading which is lower than the other two (75:25 H₂O:DMAC). In all the other cases the first reading is higher, sometimes much higher, than the other two. Although this effect is hardly statistical, it was sufficiently frequent to lead us to the conjecture that possibly the last two readings on each sample were a reflection of the fact that they had been obtained after the sample had been squeezed between the two half cells of the resistivity tester; as found some time ago, compressing or squeezing the composites leads to lower resistivity values.

Several interesting conclusions of a general nature can be drawn from the results of Table 1.

If one considers only the left column under "Specific Resistivity" it is found that resistivity values approaching the desired 150 ohms can be found sparsely in the water:DMSO extractants, consistently in two of the three dioxane-containing mixtures and in one of the acetic-acid-containing mixtures. On the other hand, the resistivity column on the right shows favorable resistivity values for all main mixtures except for those containing DMAC. It must be noted that the thickness of the samples is taken into account when calculating specific resistivity. The 25:75 H₂O: dioxane mixture values are so close to the blank value that, except in one case, it was not possible to calculate the resistivity using the relatively coarse tester used.

No particular trend was observed involving resistivity as a function of time of extraction. The five thickness readings obtained for each sample are generally relatively close to each other; the occasional high value was most likely taken close to one of the edges of the sample where chances of the mix tunneling down along the edge are greatest. In this case also no correlation was observed between time of extraction and resulting sample thicknesses.

In summary, this experiment has shown that the extracting efficiency of the twelve solutions tested ranges as follows: water:dioxane → water:DMSO → water:acetic acid → water:DMAC in decreasing order of efficiency, as shown by the resistivity values obtained. On the other hand, time of immersion in all the twelve solutions does not seem to affect strongly either specific resistivity or thickness of coated and extracted samples. It would seem that, at least for the system investigated here involving polysulfone binder, 25:75 water:dioxane is the most favorable extracting medium.

It must, however, be noted that air-dried (R.T.) Webril tape samples (total thickness after drying: 3.0 mils), gave resistivity values as low as those encountered in the water:dioxane case. PUDO 193 cellophane gave specific resistivity values of the same order of magnitude. Comparison between the "resistivity values" of the water:dioxane, air-dried and PUDO cellophane samples is, however, meaningless at the moment because of the relative insensitivity of the tester used.

Figures 1 to 23 indicate graphically the time required by 40% KOH to diffuse through the experimental composites mentioned above (no previous KOH soaking was used for these samples). This parameter is taken to show the presence of gross defects in the membranes and to indicate whether ion transfer is too slow for the required application.

Three classes of conditions observable from the results obtained are distinguished:

1. Fast diffusion. This is the predictable case of bare Webril, and the case of the samples extracted in 25:75 H₂O:dioxane (30, 120 and 360 minutes extraction, Figs. 1, 13, 14, 15). These samples, incidentally, gave the best resistivity values, but they also were relatively thin, which might account partially for their favorable properties; a borderline case is given by a sample extracted in 25:75 H₂O:DMSO (30 minutes extraction, 3.8 mils average thickness, Fig. 9).

2. Medium diffusion. In this case a pH near or above 13 is generally not reached in less than about 30 minutes of diffusion. These cases and the one above are characterized by a rapid rise of pH at the beginning of the experiment, followed by a rapidly flattening curve. The relative slope and heights are influenced by the thickness of the samples; the curves exhibiting this behavior are shown in Figs. 2, 3, 4, 5, 8, 10, 12, 20, 23. The air-dried sample falls within this category.
3. Slow diffusion. All these curves are characterized by a more or less prolonged initiation period (Figs. 6, 7, 11, 16, 17, 18, 19, 21, 22); generally after 30 minutes the pH readings do not go much above 10. The curve depicted on Fig. 19 (6 hours in 75:25 H₂O:dioxane, 3.08 mils) indicates that in 10 to 12 minutes of exposure there is practically no electrolyte diffusion through the sample. In some cases (Figs. 18, 21, 22) there appears a small but definite initial lowering of pH in the distilled water as a function of time, followed by the usual slow slope. It must be noted that the average thickness of these samples is no higher than that of samples characterized by medium or fast diffusion. To summarize the results of the electrolyte diffusion time experiment, about 20% of all the samples allow relatively fast electrolyte diffusion, the rest of the samples are equally divided between medium and slow diffusion characteristics.

This experiment has shown fairly conclusively that the samples prepared in the manner described above are free of gross defects or imperfections which would allow extremely fast electrolyte diffusion (see, for example, Fig. 1 showing the equilibration of pH in both cells in less than 1.5 minute in the case of bare Webril).

The behavior of the set of samples whose characteristics fall in the "slow diffusion" indicates that the porosity in these membranes is not sufficiently high for easy electrolyte circulation. The occasional initial pH decrease may indicate CO₂ contamination which would be clearly observable during tests where pH changes take a relatively long time.

On the whole, there does not seem to be a clear correlation between electrolyte diffusion and extraction times or solutions. The group of samples with fast electrolyte diffusion is also the one with best resistivity values. The samples falling in the "medium diffusion" category have diffusion characteristics which are still favorable, even though some of these samples may not exhibit promising specific resistivity characteristics. The "slow diffusion" samples are probably too impervious to electrolyte passage.

3.4.2 Bio Rad Zirconia - Containing Formulations

The procedure followed in this experiment is the same as set forth above. Samples of Webril tape were hand coated with a mixture consisting of 80 grams of vacuum oven dried Bio Rad zirconia (20-50 mesh) and 460 g of polysulfone:DMAC solution (15% solids, oxide: PS ratio = 20:15). This mixture had previously been ball-milled for at least 70 hours. The shimstock knife blade gap was again set at 3 mils.

Table II summarizes the results of the extraction time experiment in terms of specific resistivity. Here again there are two specific resistivity columns, to underline the effect of pressing the separator samples during the resistivity readings.

A comparison between Tables I and II readily indicates that the use of relatively coarse Bio Rad oxide results in poorer overall resistivities. Only in 25:75 water: dioxane do reasonable resistivity values appear in both columns. In the great majority of the other cases the resistivity values are very high, at times beyond the capacity of the meter. It should be noted here that, while on Table I (Report

No. 12) the value of the KOH blank must be subtracted from the unpressed resistance values to obtain the actual sample resistivities, in Table II this has already been done. Again, thicknesses of samples are reasonably consistent. The resistivity values of samples made the same way as mentioned above but air-dried rather than extracted are again low (avg. 44 ohms-cm, see Report No. 12).

It is possible that air or heat drying would be the best method for obtaining reproducibly low resistivity results.

In this experiment as well it was not possible to distinguish a reliable trend involving time of immersion and resistivity values, except in the 25:75 water:dioxane test, in which longer extractions appear to give lower resistivity values.

Figures 24 to 28 inclusive show graphically the degree of tightness of some separators, as indicated by the time of diffusion of 40% KOH through each experimental membrane. The air dried sample exhibits a relatively fast diffusion time with no apparent induction period (Fig. 24). The next three graphs (Figs. 25, 26 and 27) indicate that the high resistivity values shown in Table II for the samples tested are accompanied by elevated resistance to the flow of KOH. For this reason, pH readings were next taken only for the 25:75 H₂O:dioxane use, as shown in Fig. 28. Interpretation of these curves generally shows an agreement with the corresponding resistivity values, i.e., the longer the extraction time the greater the permeability to KOH.

In conclusion, the relatively large particle size of Bio Rad zirconia (20-50 mesh) appears to militate against the low resistivity values and high alkali diffusion values required of satisfactory battery separators, at least under the conditions of our experiment. However, a 25:75 water:dioxane extractant solution does yield separators of acceptable quality.

4. Continuous Runs

4.1 TAM Zirconia: PS = 20:15

Dried TAM zirconia was used in this continuous run; the mix, which was light brown because of the color attained by the zirconia during vacuum-oven drying, was ball-milled for 48 hours before use. The extracting solution used was 1:2 H₂O:dioxane. The gap at the knife was nominally 3 mils and that at the compression rollers was 2 mils initially. The coating speed was 1 ft/min. The bare Webril tape coated well under these conditions; however, pinholes were visible against the heating lamps located under the tape between the extractant bath and the take-up rollers.

After taking samples from this section of the run, the coated tape was coated again with a knife gap of 5 mils and a roller gap of 3 mils. The latter resulted too narrow, so that it was widened approximately to 4 mils. The second treatment removed all the pinholes but it may have weakened the original coat by contact of the latter with the solvent in the mix during the second addition. The average specific resistance of the first batch of coated tape (3 mils, one coat) was 120 ohms-cm, and the average thickness was 2.1 mils. The average specific resistance of the double coated sample was 190 ohms-cm and the average thickness 3.3 mils. Figs. 29 and 30 give the diffusion time of 40% KOH through both samples, indicating that the singly coated separator is more permeable than the doubly coated one, as is reasonable. However, the single pass sample may be too porous, as shown by pH values.

It was concluded that making a continuous run at a knife gap of 4 mils, with a roller gap of 3 mils, would give pinhole free separator material.

4.2 Bio Rad (20:50 Mesh): PS = 30:15

Another continuous run was made on 2.5 inch wide Webril tape. The coating mixture consisted of 120 g of dry Bio Rad zirconia (20-50 mesh) ball milled with 460 g of polysulfone:DMAC solution (25 g of PS

to 100 cc of DMAC, 2:1 oxide:PS ratio). The knife gap was 4 mils and the gap at the roller was initially 3 mils. It was found in the course of the experiment that the roller gap was too narrow and caused the tape to tear; therefore the top roller was allowed just to press on the coated tape by gravity. This experiment investigated the effect of (1) higher oxide loading, (2) different coating speeds, (3) different extractant ratios, and (4) greater knife gap than used in any of the other experiments carried out so far. The coating speed range was from 1 to 6 feet per minute and the extractant consisted of 1 volume of water to three volumes of dioxane. Four heating lamps were situated near the coated tape, and they effectively dried the coated tape well enough so that the rolled up tape did not block. The speed for drying was inversely proportional to the coating speed, as one might expect. Again, it was noted that it is imperative that the material in the feed tanks be agitated periodically to prevent the oxide from settling out.

At the end of the run the coated separator material was divided into sections coated at different speeds and samples were taken from each section. These samples were tested for specific resistivity and time of electrolyte diffusion as usual. Table III summarizes some of these results.

The coated tape obtained at a speed of about 3 feet per minute seemed best as far as homogeneity and appearance of coating. At one foot and at two feet per minute the coating mixture had time to form runs down along the edges of the tape, starting from the reservoir on the knife blades - this ruined the homogeneity of the tape. The six feet per minute coating speed was too fast, the quantity of the coating mixture in the reservoir not being able to keep up with the passage of the Webril tape. This resulted in occasionally starved sections.

The data shown in Table III indicate that specific resistivity values of the Webril tape coated at a number of speeds all fall considerably below the specific resistivity required for acceptable separators. While no trend is noticeable in the resistivity values,

the average thickness of the coated tape appears to decrease with increasing coating speed. Again, the hand coated, air dried samples exhibit favorable values of thickness and resistivity.

Fig. 31 shows the behavior of separator samples, taken at different coating speeds from this continuous run, in contact with 40% KOH. The air-dried (1-3 hours) sample exhibits the fastest and highest electrolyte diffusion time; if one neglects the anomalous behavior of the sample coated at 3.16 ft/min, the lowest diffusion is shown by the sample taken at 1 ft/min. The final pH values attained by the samples taken at the other speeds fall within 1 pH unit and their pH increase rates are similar.

III. GENERAL CONCLUSIONS

The results of the investigations carried on until now may be summarized as follows:

- a. Dioxane - containing extracting solutions, especially the ones in which the dioxane is the preponderant ingredient, yield battery separator composites which have so far shown the best resistivity and ion diffusion-values. This indicates that the pore size and distribution of these materials are fairly uniform.
- b. Except in one or two cases, time of immersion does not seem to be an important factor in the preparation of satisfactory separator material. This may be due to the fact that the coagulation and solvent leaching actions are fast and simultaneous.
- c. Air-dried samples coated with the formulations used in the investigation of the effects of immersion times consistently show good resistivity and KOH diffusion values.

- d. Continuous runs at speeds of about 3 feet per minute, using a 1:3 water:dioxane extractant ratio (by volume), a 2:1 zirconia: PS ratio and a 4 mil knife blade gap, are capable of yielding reproducibly separator materials which are acceptable from the specific resistivity, thickness and electrolyte diffusion points of view.
- e. Care must be taken in drawing conclusions from comparisons between hand made samples and samples obtained from continuous runs. The properties of the latter are usually and obviously more reproducible than those of the hand made samples. However, the investigations involving series of hand-made samples are valuable for having indicated trends in solvent leaching characteristics of extracting solutions.


IV. FUTURE WORK

Higher loadings of zirconia in the regular PS formulations will be explored.

The possibility of doing away with the precipitating and extracting solutions and of using heating instead to eliminate the solvent and to cause porosity during continuous runs, will be explored.

Procedures will be set up for the polarographic measurement of silver and zinc ions diffusing through experimental battery separator composites.

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Surface Coatings

TABLE 1

Sample No.	Immersion Time, Min.	Extractant Composition by Volume	KOH Blank, Ohms	Unpressed Resistance of Sample Plus KOH, Ohms	Specific Resistivity		Thickness, Mils (before testing)	
					Avg. of 3 Resist.	Avg. of 2 last Resist.	Individual	Avg.
C58-4-3-1	1	50:50 DMAC:H ₂ O	160	185, 170, 173	567	388	3.5/4.1/3.6/3.6/3.8	3.52
	30			215, 195, 172	1230	740	4.3/4.0/3.7/4.2/3.0	3.84
	120			183, 172, 169	600	400	2.5/3.1/3.4/3.1/3.2	3.08
	360			178, 175, 190	780	800	3.3/3.6/3.3/2.1/3.5	3.36
C58-4-3-2	1	25:75 H ₂ O:DMAC	158	182, 168, 168	600	400	3.2/3.3/2.8/2.9/3.4	3.12
	30			200, 176, 170	930	550	3.0/2.4/2.7/3.0/2.6	2.94
	120			170, 170, 160	390	300	2.4/3.1/2.5/2.9/3.0	2.88
	360			200, 170, 170	720	390	3.8/5.0/3.9/3.2/3.2	3.82
C58-4-3-3	1	75:25 H ₂ O:DMAC	165	170, 190, 175	440	575	3.5/3.5/3.2/2.5/4.7	3.68
	30			160, 170, 162	---	---	4.0/3.8/3.4/3.4/3.4	3.66
	120			190, 195, 185	1110	1110	2.4/3.0/2.7/2.9/3.0	2.8
	360			180, 175, 170	375	280	2.8/4.0/3.1/3.0/3.7	3.32
C58-4-3-4	1	50:50 H ₂ O:DMSO	168	195, 168, 165	330	---	3.2/2.4/3.1/3.0/2.8	3.00
	30			205, 170, 170	480	68	3.0/3.5/3.5/4.3/3.4	3.64
	120			168, 168, 170	41	41	4.1/2.4/2.8/3.0/3.1	3.02
	360			178, 170, 168	184	37	3.2/3.9/3.2/3.1/3.5	3.38
C58-4-3-5	1	25:75 H ₂ O:DMSO	165	172, 168, 168	175	87	2.0/2.5/2.2/2.7/2.5	2.85
	30			190, 175, 175	490	328	4.4/3.6/3.7/3.9/3.4	3.80
	120			200, 170, 168	530	141	3.5/3.3/4.0/3.4/3.5	3.50
	360			180, 165, 168	243	73	2.5/2.6/2.3/7.8/2.6	2.56
C58-4-3-6	1	75:25 H ₂ O:DMSO	162	228, 168, 168	1170	270	3.1/2.5/2.7/2.8/2.7	2.76
	30			172, 168, 168	290	218	3.2/4.3/3.3/2.0/3.5	3.46
	120			190, 165, 170	420	196	3.4/4.5/3.2/3.6/4.4	3.82
	360			168, 165, 165	160	133	2.7/2.0/2.7/3.1/2.5	2.78

TABLE 1 (Cont'd)

Sample No.	Immersion Time, Min.	Extractant Composition by Volume	KOH Blank, Ohms	Unpressed Resistance of Sample Plus KOH, Ohms	Specific Resistivity		Avg. of 2 last Resist.	Individual	Thickness, Mils (before testing)	Avg.
					Avg. of 3 Resist.	Ohm cm				
C58-4-3-7	1	50:50 H ₂ O:Dioxane	160	300, 185, 175	2120	700		3.6/3.2/3.2/3.5/3.3		3.36
	30			218, 168, 165	930	300		3.3/2.8/3.3/3.6/3.0	3.20	
	120			230, 165, 168	1050	316		3.4/3.3/3.1/3.0/2.6	3.14	
	360			330, 168, 168	2308	306		3.4/3.1/3.1/3.2/3.4	3.24	
C58-4-3-8	1	25:75 H ₂ O:Dioxane	165	165, 165, 165	---	---		2.4/2.4/2.1/2.2/2.6		2.34
	30			165, 165, 165	---	---		2.5/2.6/2.7/2.5/2.6	2.50	
	120			165, 165, 165	---	---		2.9/2.6/2.8/2.7/2.6	2.72	
	360			167, 165, 165	40	40		7.9/3.1/3.0/3.1/3.0	3.02	
C58-4-3-9	1	75:25 H ₂ O:Dioxane	163	180, 165, 165	288	85		2.6/3.3/2.9/2.9/3.4		3.02
	30			168, 163, 167	115	77		3.4/3.4/2.1/3.6/3.0	3.22	
	120			178, 163, 165	203	42		2.9/3.2/2.9/3.0/3.3	3.06	
	360			220, 170, 163	1600	161		3.0/3.2/3.0/3.1/3.1	3.08	
C58-4-3-10	1	50:50 H ₂ O:Ac. Acid	165	215, 177, 102	1170	744		3.0/3.8/2.9/3.0/3.2		3.18
	30			193, 168, 170	485	161		3.1/3.5/3.1/3.1/3.6	3.08	
	120			198, 183, 183	875	685		2.3/3.2/3.3/3.3/3.3	3.28	
	360			202, 170, 175	600	265		4.0/3.2/3.2/3.2/3.0	3.54	
C58-4-3-11	1	25:75 H ₂ O:Ac. Acid	167	183, 188, 167	530	450		3.1/3.0/3.0/3.1/3.1		3.06
	30			177, 168, 171	210	165		0.1/3.0/3.0/2.7/3.0	2.86	
	120			195, 180, 178	660	467		3.3/3.2/3.3/3.0/3.2	3.2	
	360			205, 170, 173	590	165		3.9/3.1/3.8/3.0/3.1	3.38	
C58-4-3-12	1	75:25 H ₂ O:Ac. Acid	167	172, 168, 168	81	41		3.2/3.2/3.0/2.9/3.1		3.08
	30			185, 179, 168	137	21		3.4/3.7/3.6/3.6/3.7	3.7	
	120			192, 168, 177	515	280		2.9/3.0/2.8/2.8/3.0	2.9	
	360			218, 170, 168	660	73		4.0/3.6/3.5/3.0/3.6	3.4	

TABLE II

Sample No.	Immersion Time, Min.	Extractant Composition by Volume	KOH Blank, Ohms	Unpressed		Specific Resistivity		Avg.
				Resistance of Sample, Ohms	Avg. of 3 Resist.	Ohm cm	Thickness, Mils (before testing)	
C58-12-2-1	1	H ₂ O:DMAC 50:50	170	28, 32, 25	120	120	3.0/2.9/3.1/3.0/2.7	2.9
	30			140, 55, 83	400	296	3.1/2.9/3.0/2.7/3.0	2.9
	120			108, 32, 108	364	311	3.1/2.7/2.7/2.8/2.8	2.8
	360			115, 48, 60	273	198	2.9/3.3/3.4/2.6/3.0	3.4
C58-12-2-2	1	H ₂ O:DMAC 25:75	170	330+, 125, 58	>1500	410	2.8/3.1/2.5/2.8/2.6	2.75
	30			330+, 28, 133	>1500	342	3.3/3.3/2.0/2.5/2.7	2.9
	120			330+, 160, 105	>1500	567	3.1/3.0/2.8/2.6/2.8	2.9
	360			295, 330+, 330+	>1500	>1500	3.0/3.0/3.8/3.9/3.3	3.2
C58-12-2-3	1	H ₂ O:DMAC 75:25	170	300, 27, 82	513	207	3.8/3.3/3.1/3.1/3.0	3.3
	30			300, 22, 38	480	120	3.1/3.3/3.3/2.8/3.1	3.1
	120			142, 35, 48	327	182	2.8/2.9/2.8/2.6/3.0	2.85
	360			330+, 108, 65	>1500	320	3.4/3.4/3.2/3.0/3.2	3.34
C58-12-2-4	1	H ₂ O:DMSO 50:50	178	322+, 247, 147	>1500	767	3.4/3.3/3.4/2.9/3.0	3.2
	30			322+, 322+, 322+	>1500	>1500	3.8/3.3/3.8/3.2/3.2	3.5
	120			52, 182, 242	685	920	2.7/3.3/3.0/2.7/2.8	2.9
	360			20, 32, 115	228	303	3.0/2.8/3.1/3.0/3.0	3.0
C58-12-2-5	1	H ₂ O:DMSO 25:75	178	322+, 322+, 322+	>1500	>1500	2.9/2.8/2.7/2.7/2.5	2.6
	30			322+, 322+, 322+	>1500	>1500	3.0/3.2/3.0/2.7/3.1	3.0
	120			322+, 95, 322+	>1500	>1500	3.3/3.0/3.2/3.2/2.8	3.1
	360			322+, 322+, 322+	>1500	>1500	2.9/3.0/2.8/3.0/2.9	2.9
C58-12-2-6	1	H ₂ O:DMSO 75:25	195	305+, 305+, 305+	>1500	>1500	3.0/3.4/3.0/3.1/3.1	3.1
	30			305+, 87, 180	>1500	535	2.9/3.1/2.9/2.8/3.1	3.0
	120			255, 5, 97	477	205	3.0/3.6/3.0/2.9/2.9	3.1
	360			100, 0, 0	128	---	2.9/3.9/3.2/2.7/3.1	3.2

TABLE II (Cont'd)

Sample No.	Immersion Time, Min.	Extractant Composition by Volume	KOH Blank, Ohms	Unpressed		Specific Resistivity Ohm cm		Thickness, Mils (before testing)		Avg.
				Resistance of Sample, Ohms	Avg. of 3 Resist.	Avg. of 2 last Resist.	Individual			
C58-12-2-7	1	H ₂ O: Dioxane 50:50	169	84, 266, 321+	>1500	>1500	2.9/3.0/3.1/2.9/3.0	3.0	3.15	3.1
	30			321+, 106, 131	>1500	467	3.0/3.4/3.1/3.1/3.1			
	120			249, 151, 220	830	742	3.2/2.9/3.1/3.1/3.1			
	360			321+, 119, 321+	>1500	>1500	3.0/3.1/3.1/3.1/3.1			
C58-12-2-8	1	H ₂ O:Dioxane 25:75	166	9, 14, 58	108	144	3.3/3.3/3.3/2.9/2.9	3.1	3.0	3.0
	30			14, 7, 4	33	22.8	3.0/3.0/3.0/3.0/3.2			
	120			19, 6, 9	45.6	31	3.0/3.0/3.0/3.0/3.0			
	360			11, 3, 6	28.9	18.7	3.0/2.9/3.1/3.1/2.9			
C58-12-2-9	1	H ₂ O:Dioxane 75:25	167	241, 201, 21	567	405	3.4/3.6/3.0/3.6/3.2	3.4	3.1	2.7
	30			218, 183, 38	586	447	3.2/2.9/3.2/3.2/3.1			
	120			165, 176, 100	680	638	2.7/2.9/2.7/2.6/2.8			
	360			215, 220, 333+	>1500	>1500	2.8/3.1/3.1/2.8/2.9			
C58-12-2-10	1	H ₂ O:Ac. Ac. 50:50	163	169, 189, 337+	>1500	>1500	2.8/3.0/3.0/2.8/3.0	2.9	2.9	3.1
	30			337+, 337+, 337+	>1500	>1500	2.8/3.0/3.0/2.9/3.0			
	120			289, 337+, 337+	>1500	>1500	3.0/3.0/2.9/2.9/2.9			
	360			337+, 102, 82	>1500	368	3.2/3.0/3.2/2.9/3.0			
C58-12-2-11	1	H ₂ O:Ac. Ac. 25:75	167	333+, 53, 333+	>1500	>1500	2.9/3.0/3.0/3.0/3.0	3.0	2.8	3.4
	30			333+, 33, 263	>1500	657	2.8/2.8/2.8/2.8/2.9			
	120			333+, 333+, 333+	>1500	>1500	3.6/3.5/3.3/3.2/3.3			
	360			333+, 193, 333+	>1500	>1500	3.1/3.0/3.0/2.9/2.8			
C58-12-2-12	1	H ₂ O:Ac. Ac. 75:25	167	280, 333+, 108	>1500	>1500	3.0/3.0/3.2/2.4/3.1	3.0	3.3	3.2
	30			25, 333+, 333+	>1500	>1500	3.4/3.3/3.1/3.2/3.3			
	120			228, 333+, 333+	>1500	>1500	3.3/2.9/3.3/3.1/3.2			
	360			333+, 10, 333+	>1500	>1500	3.0/3.0/3.3/3.0/2.9			

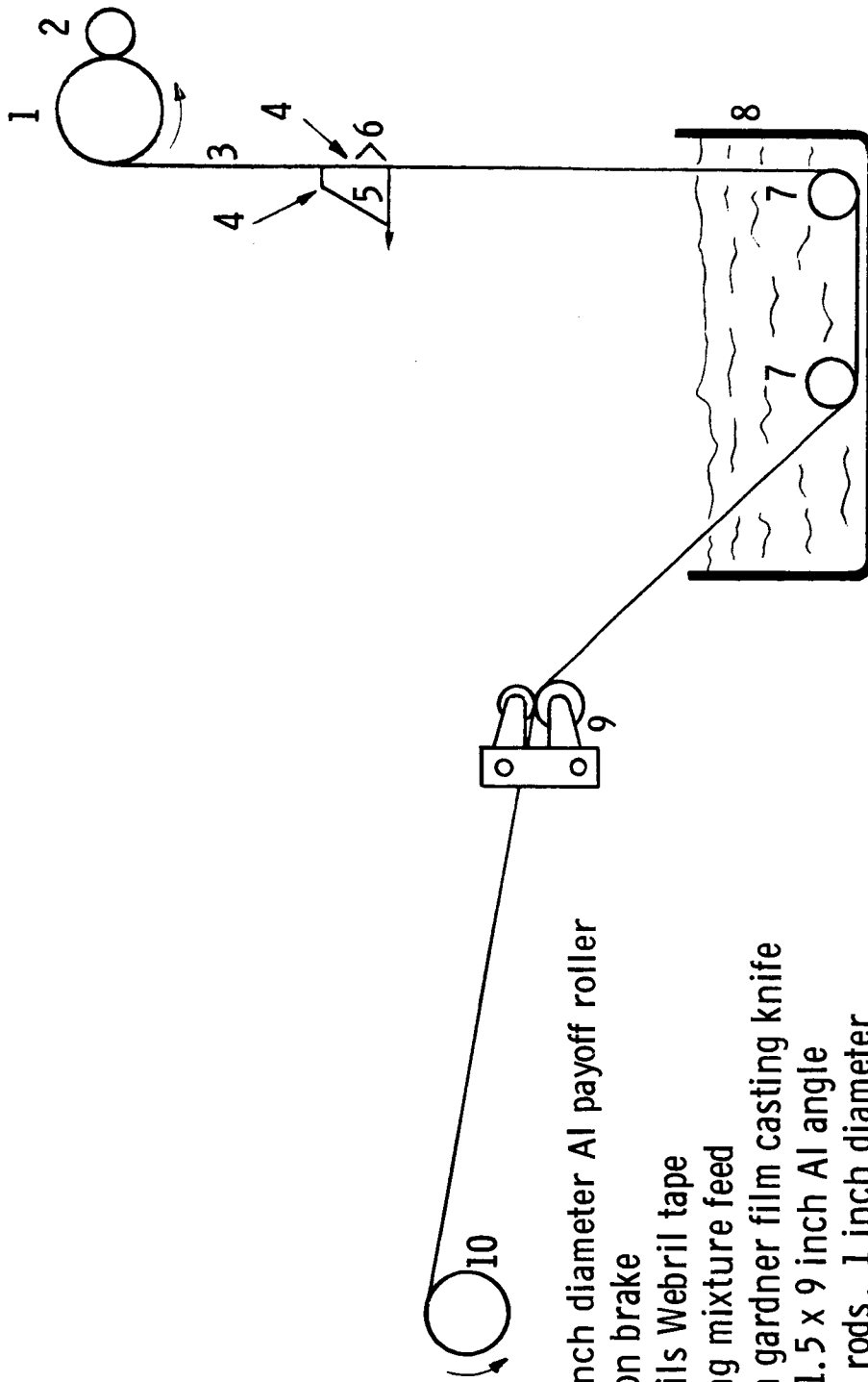
TABLE III

SUMMARY OF CONDITIONS AND RESULTS OF CONTINUOUS RUN C58-18-1

<u>Speed of Coating ft/min.</u>	<u>Average Dry Thickness, mils</u>	<u>Resistance of Unpressed Samples ohms</u>	<u>Specific Resistivity, ohms-cm</u>
1	4.0	2,2,2	62
2	3.3	2,2,2	75
3.16	3.4	2,2,2	73
3.16 (duplicate)	3.4	2,3,2	69
3.34 (begin)	3.3	1,2,2	73
3.34 (end)	3.0	2,2,2	83
6	3.2	2,2,2	73

STANDARD: AIR-DRIED, HAND COATED SAMPLES, SAME FORMULATION

3.0	2,1,1	71
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1. 3.45 inch diameter Al payoff roller
2. Friction brake
3. 1.5 mils Webril tape
4. Coating mixture feed
5. 8 inch gardner film casting knife
6. 1.5 x 1.5 x 9 inch Al angle
7. Teflon rods, 1 inch diameter
8. 18 x 12 x 6 inch tank
9. Adjustable compression rollers
10. Driven 2 inch diameter take up roller

Fig. 1A—Continuous coating machine

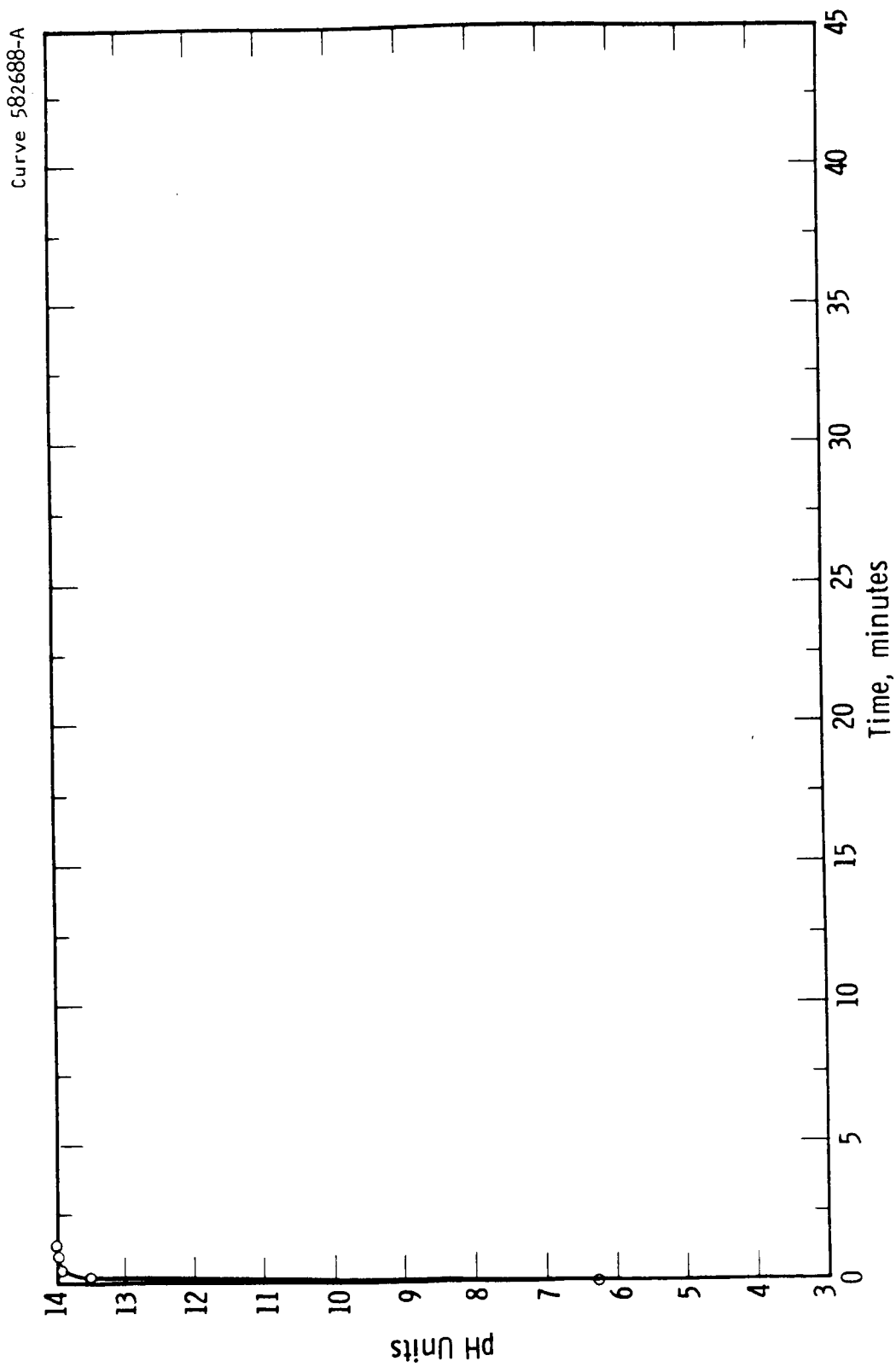


Fig. 1—Electrolyte diffusion time, 40% KOH, 26°C. Bare Webril tape, ave. thickness 1.91 mils

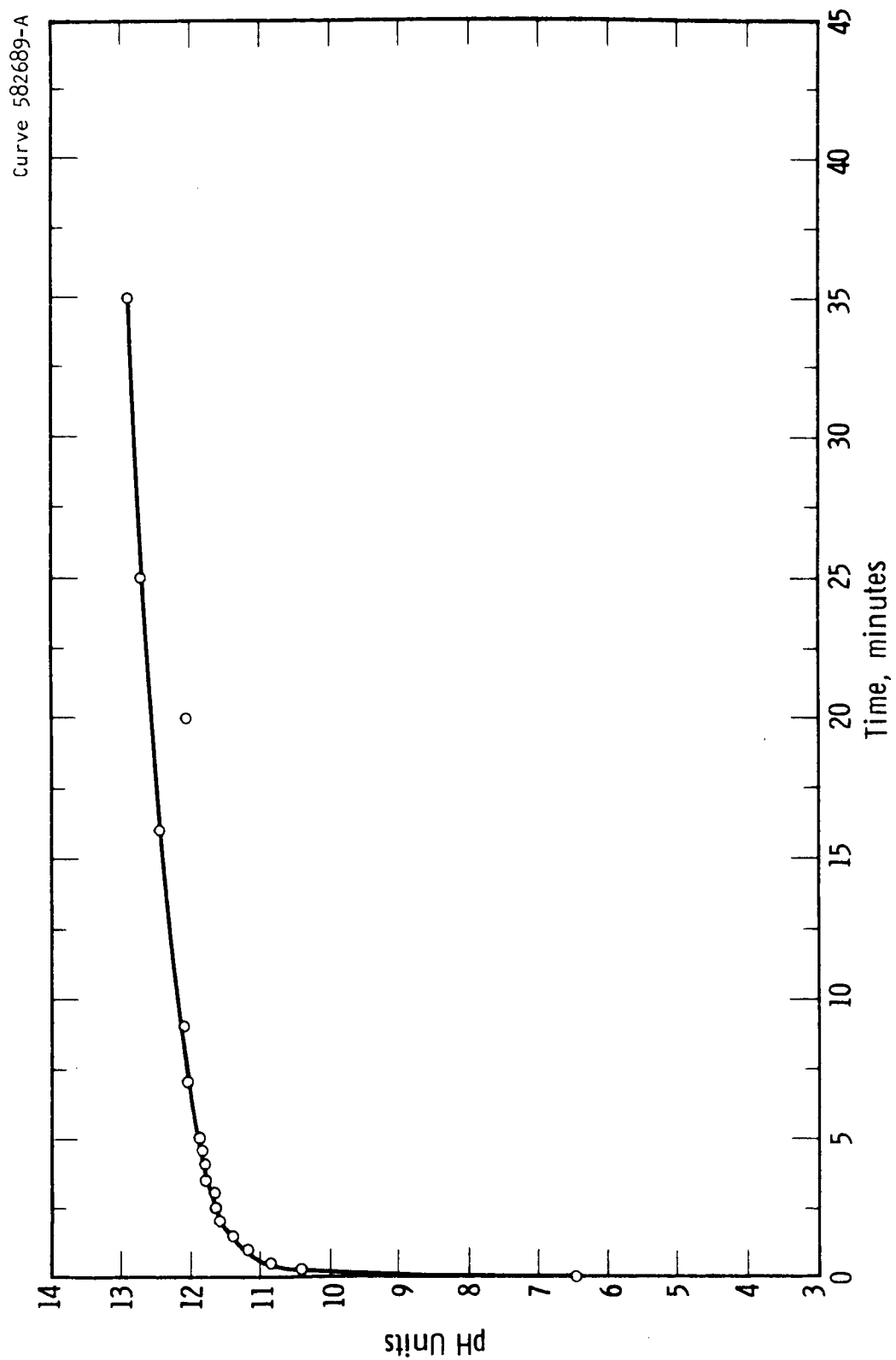


Fig. 2—Electrolyte diffusion time, 40% KOH, 26°C. PUDO 193 cellophane, ave. thickness 1 mil

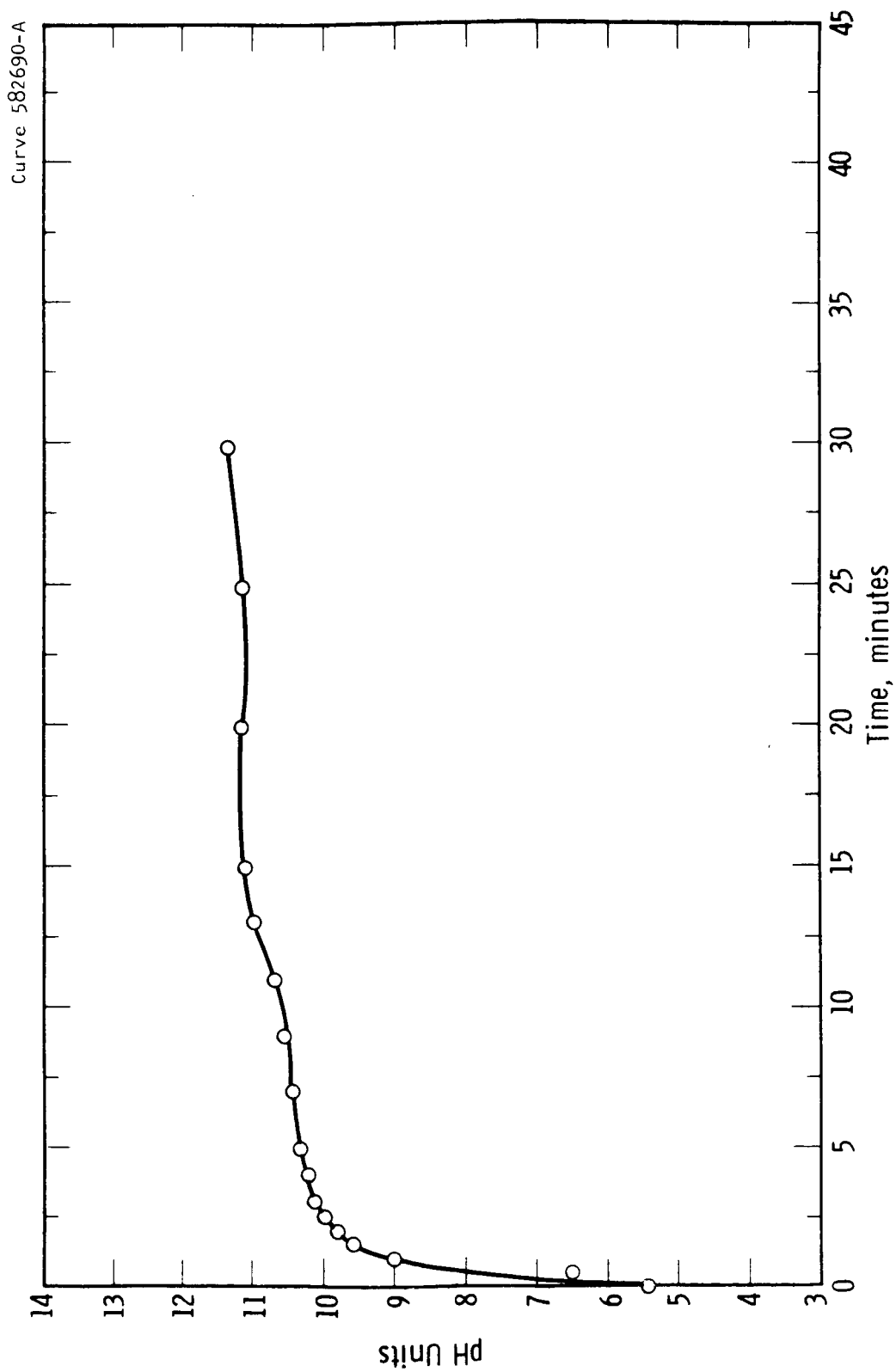


Fig. 3—Electrolyte diffusion time, 40% KOH, 26°C. Coated Webril (Zeolon H, Polysulfone), ave. thickness 3.0 mils, air dried (not extracted)

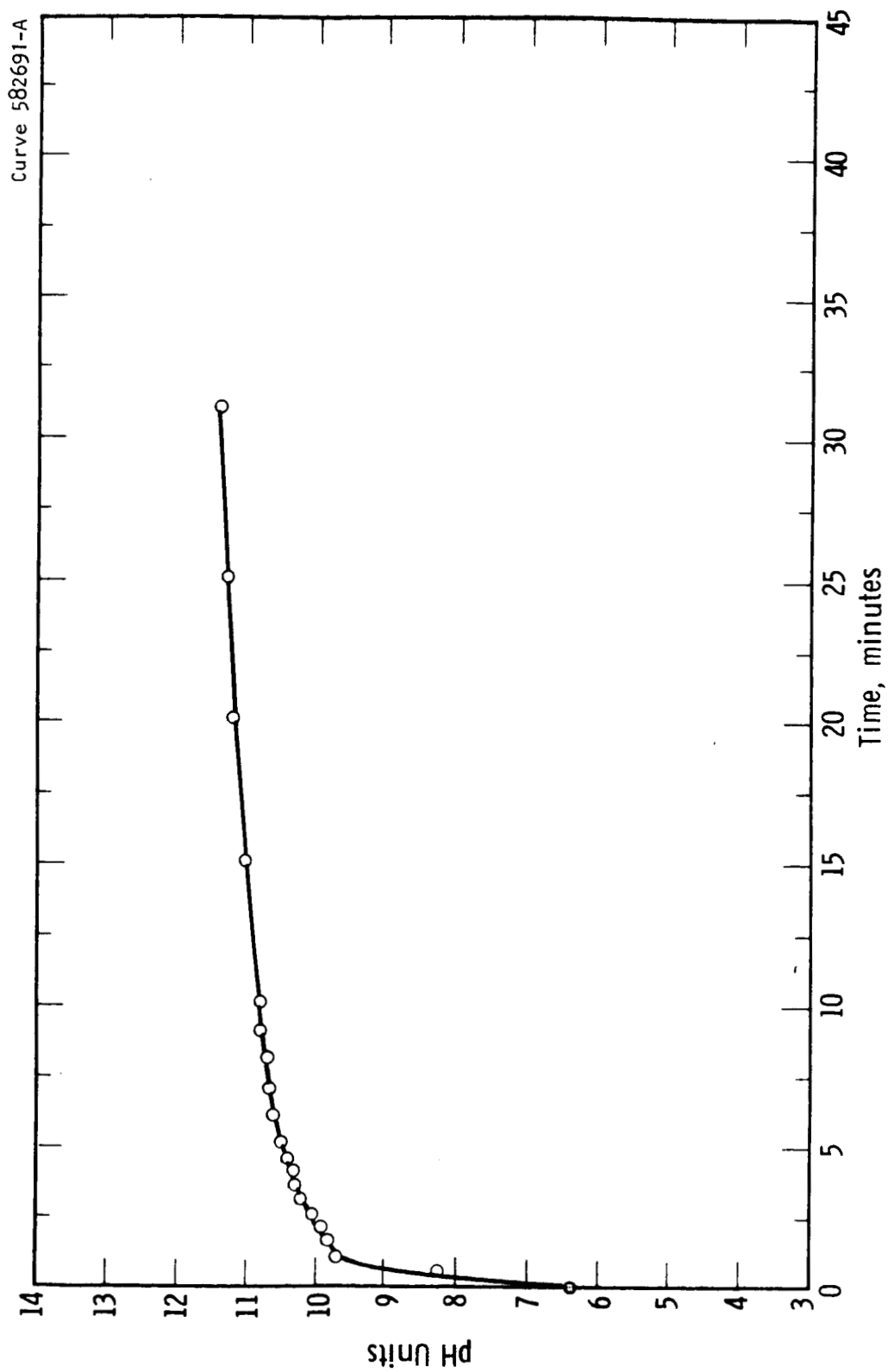


Fig. 4—Electrolyte diffusion time, 40% KOH, 26°C. Sample C58-4-3-4 extracted 1 min. in H₂O:
DMSO (50:50), ave. thickness 3.0 mils, Zeolon H : Polysulfone 20:15

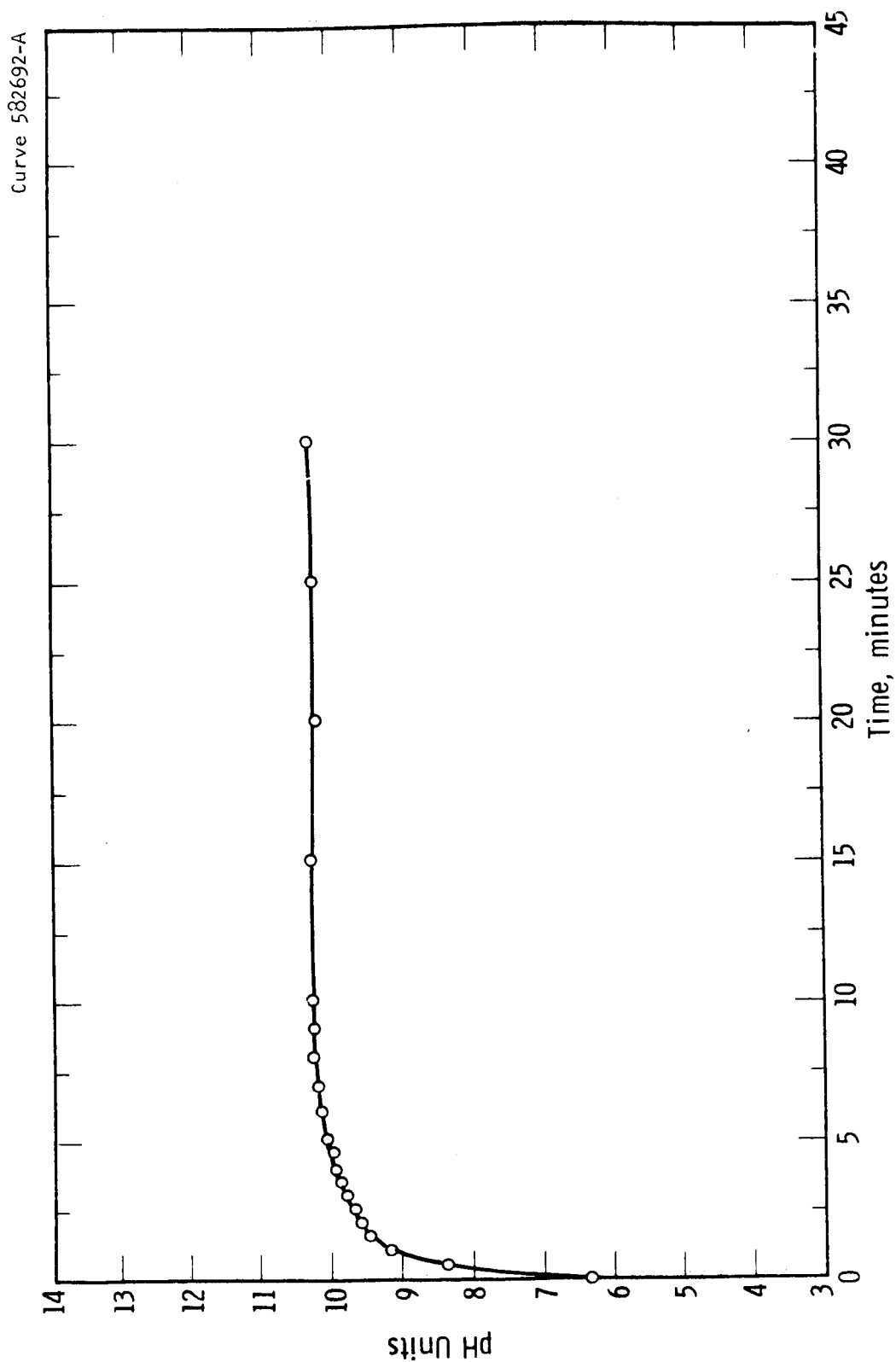


Fig. 5—Electrolyte diffusion time, 40% KOH, 26°C. Sample C58-4-3-4 extracted 30 min. in H₂O:
DMSO (50:50), ave. thickness 3.64 mils, Zeolon H : Polysulfone 20:15

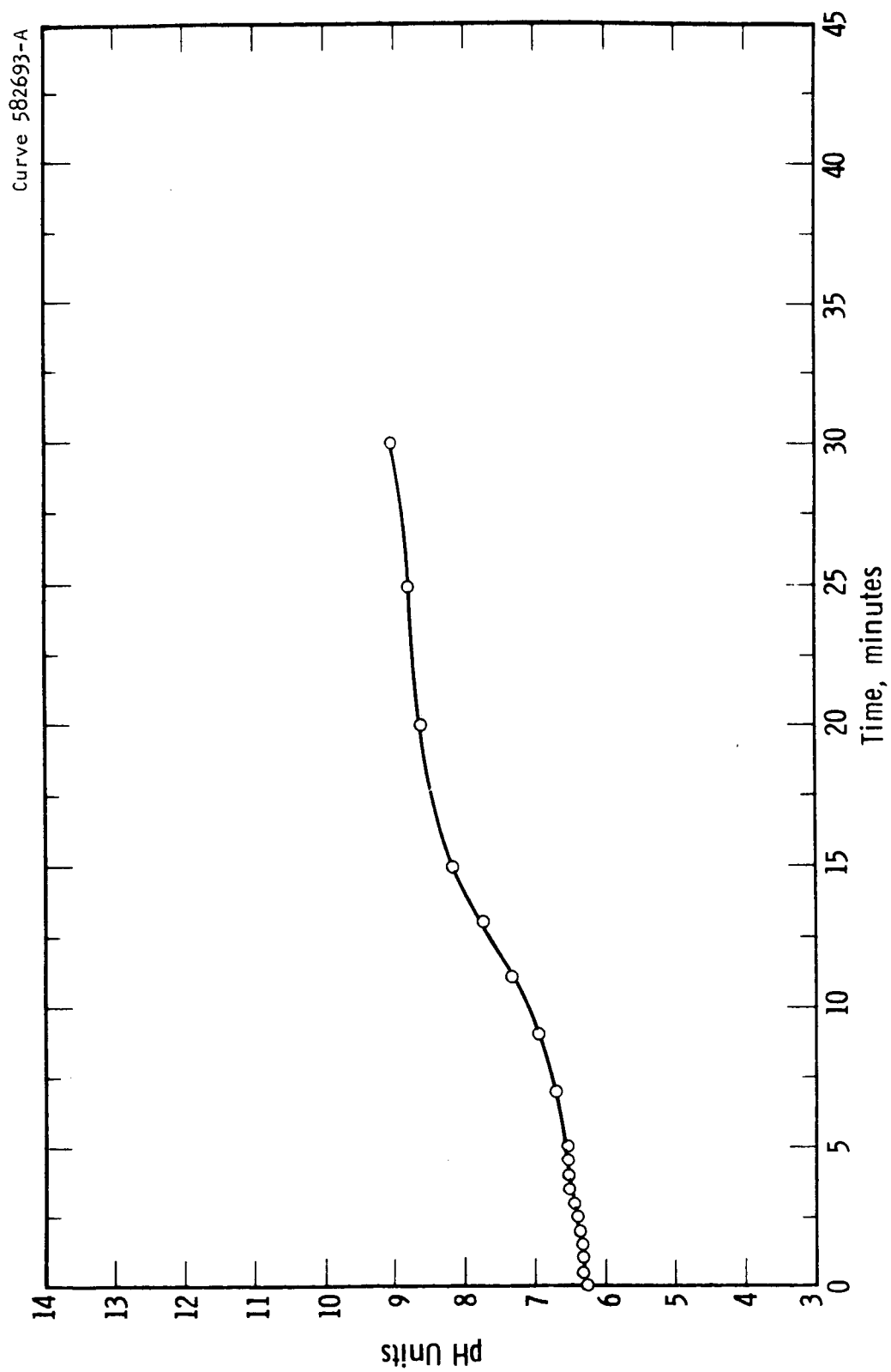


Fig. 6—Electrolyte diffusion time, 40% KOH, 26°C. Sample C58-4-3-4 extracted 2 hours in H₂O:
DMSO (50:50), ave. thickness 3.02 mils, Zeolon H : Polysulfone 20:15

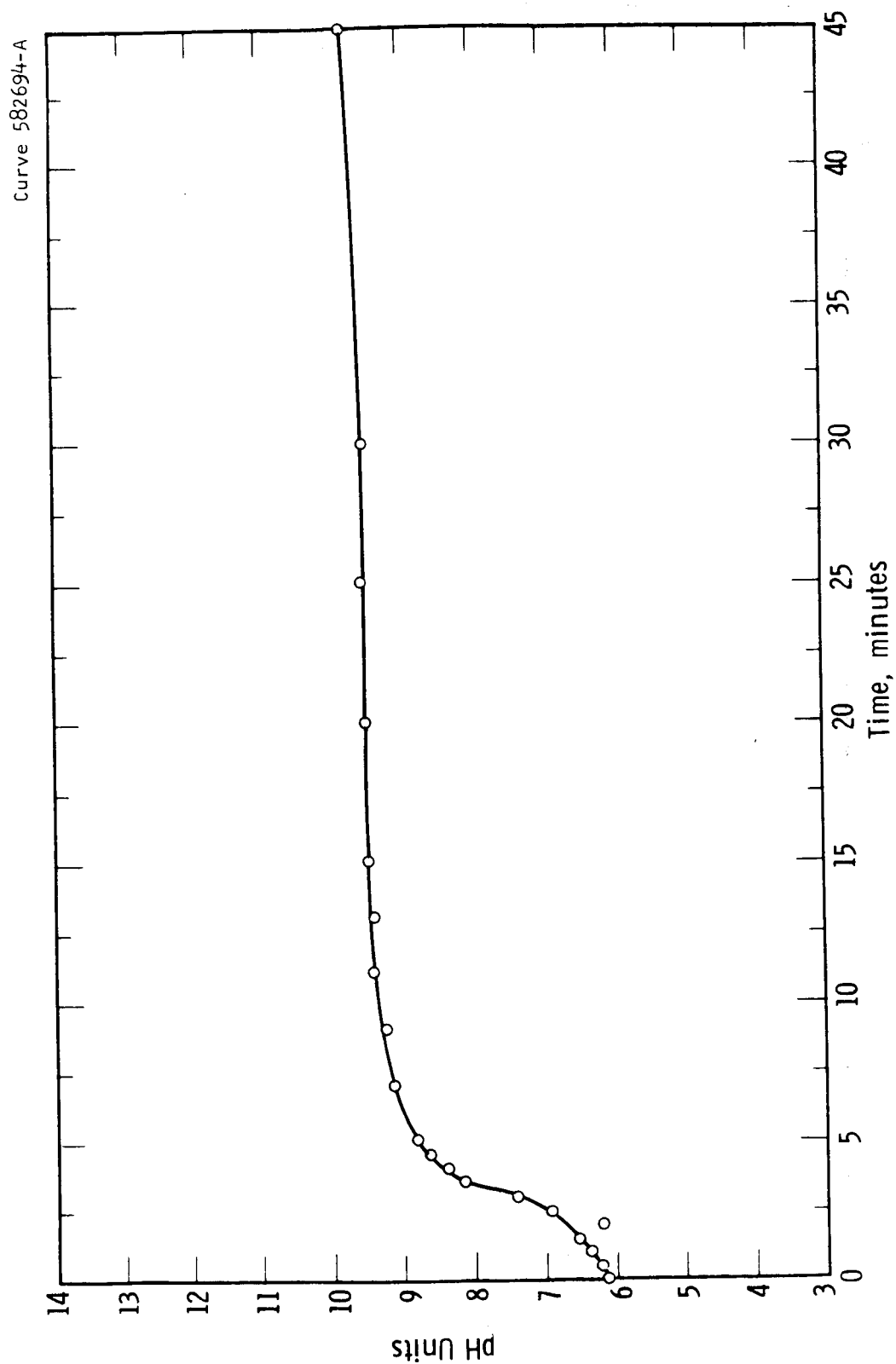


Fig. 7—Electrolyte diffusion time, 40% KOH, 26°C. Sample C58-4-3-4 extracted 6 hours in H_2O :
DMSO (50:50), ave. thickness 3.4 mils, Zeolon H : Polysulfone 20:15

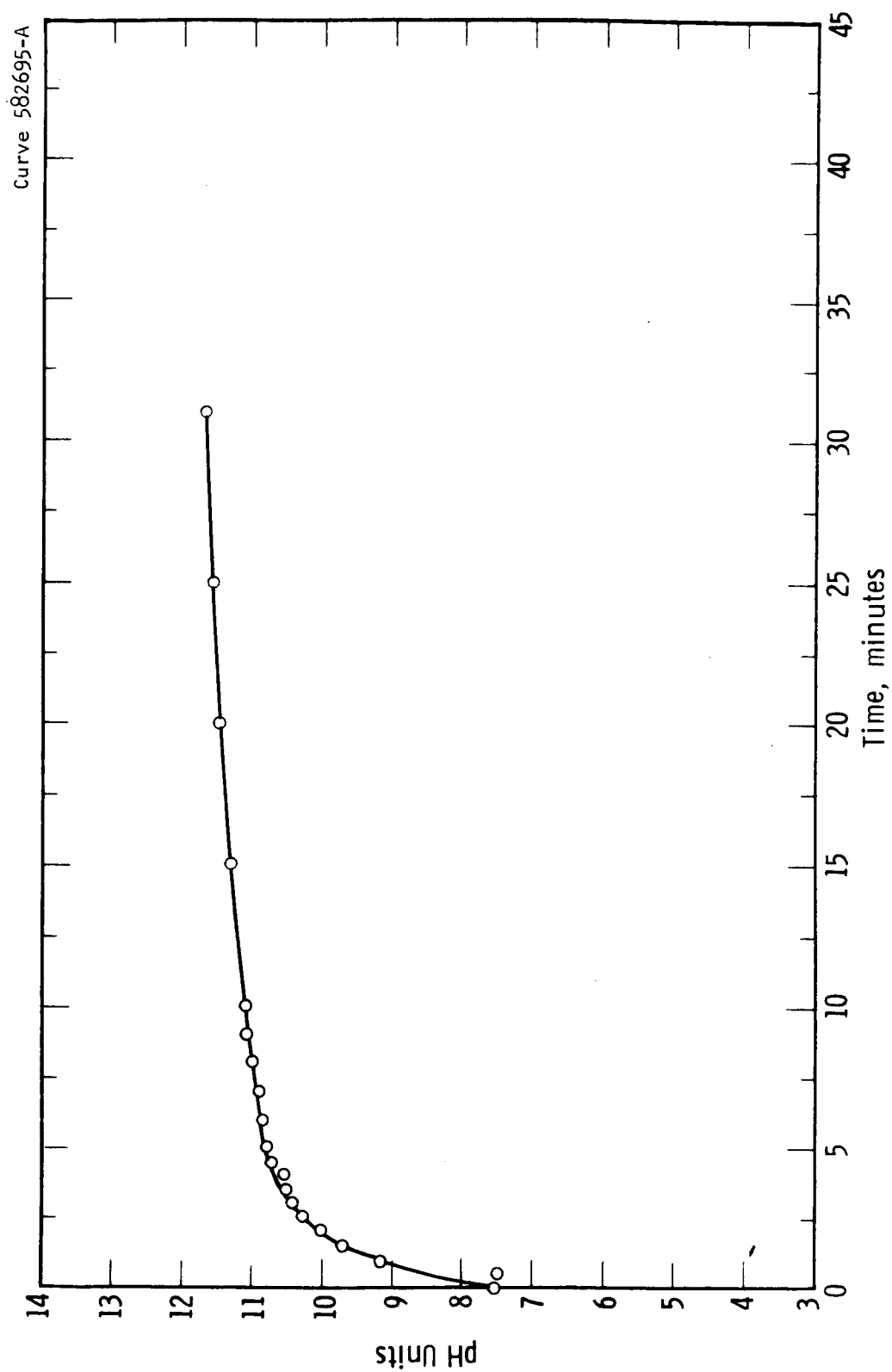


Fig. 8—Electrolyte diffusion time, 40% KOH, 26°C. Sample C58-4-3-5 extracted 1 min. in H₂O:
DMSO (25:75), ave. thickness 2.85 mils, Zeolon H : Polysulfone 20:15

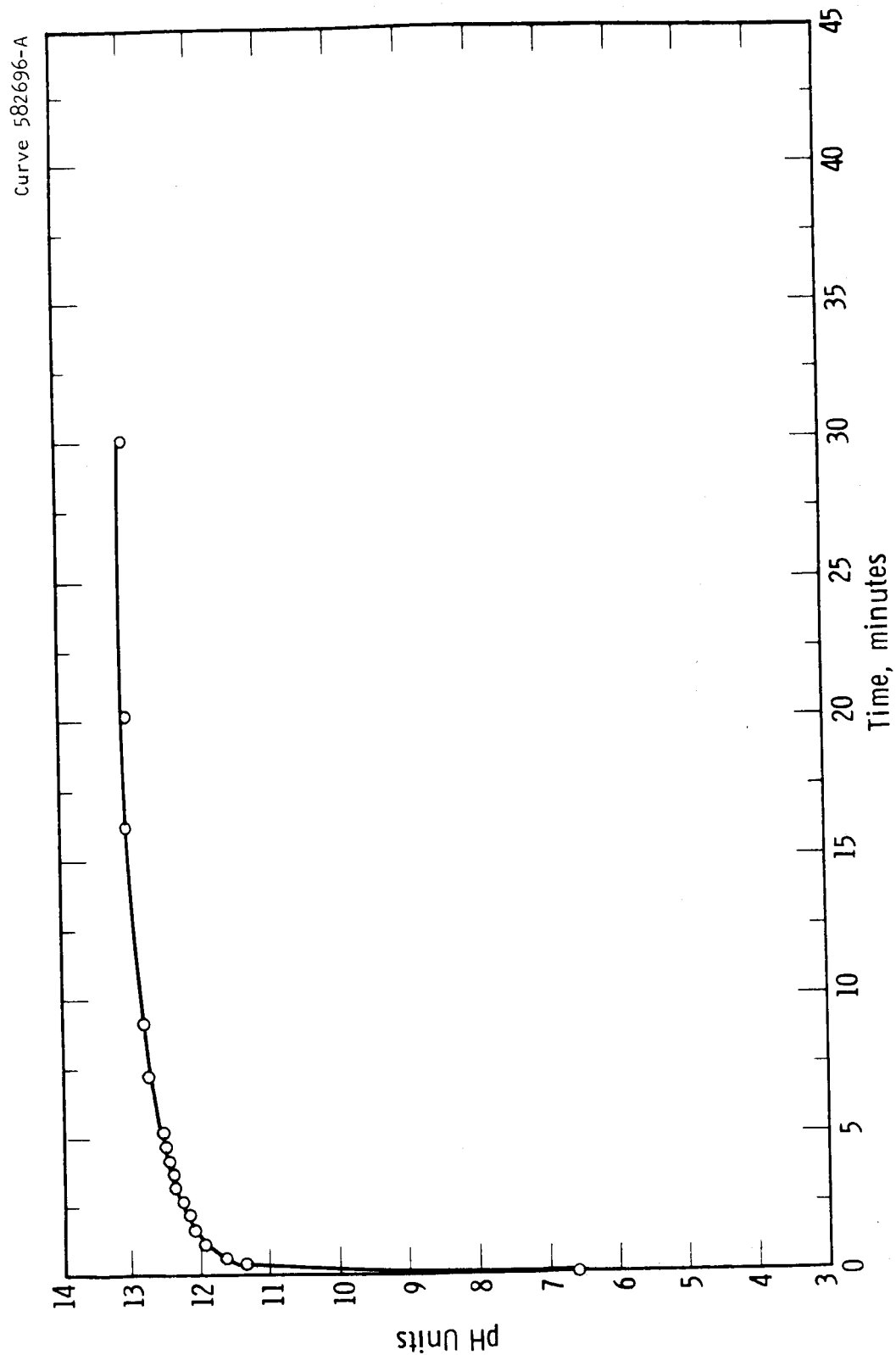


Fig. 9--Electrolyte diffusion time, 40% KOH, 26°C. Sample C58-4-3-5 extracted 30 min. in H₂O:
DMSO (25:75), ave. thickness 3.8 mils, Zeolon H : Polysulfone 20:15

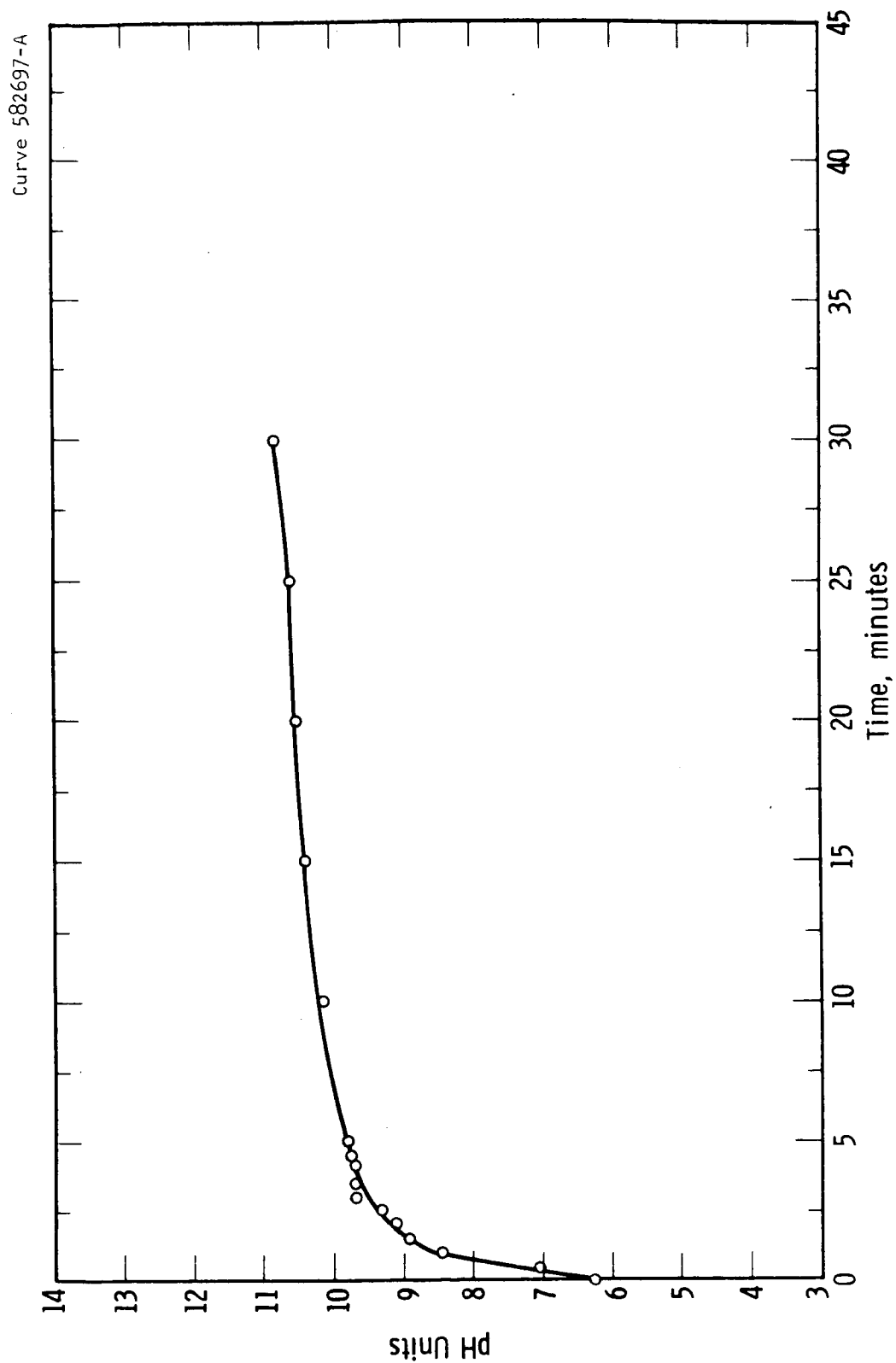


Fig. 10—Electrolyte diffusion time, 40% KOH, 26°C. Sample C58-4-3-5 extracted 2 hours in H₂O:
DMSO (25:75), ave. thickness 3.54 mils, Zeolon H : Polysulfone 20:15

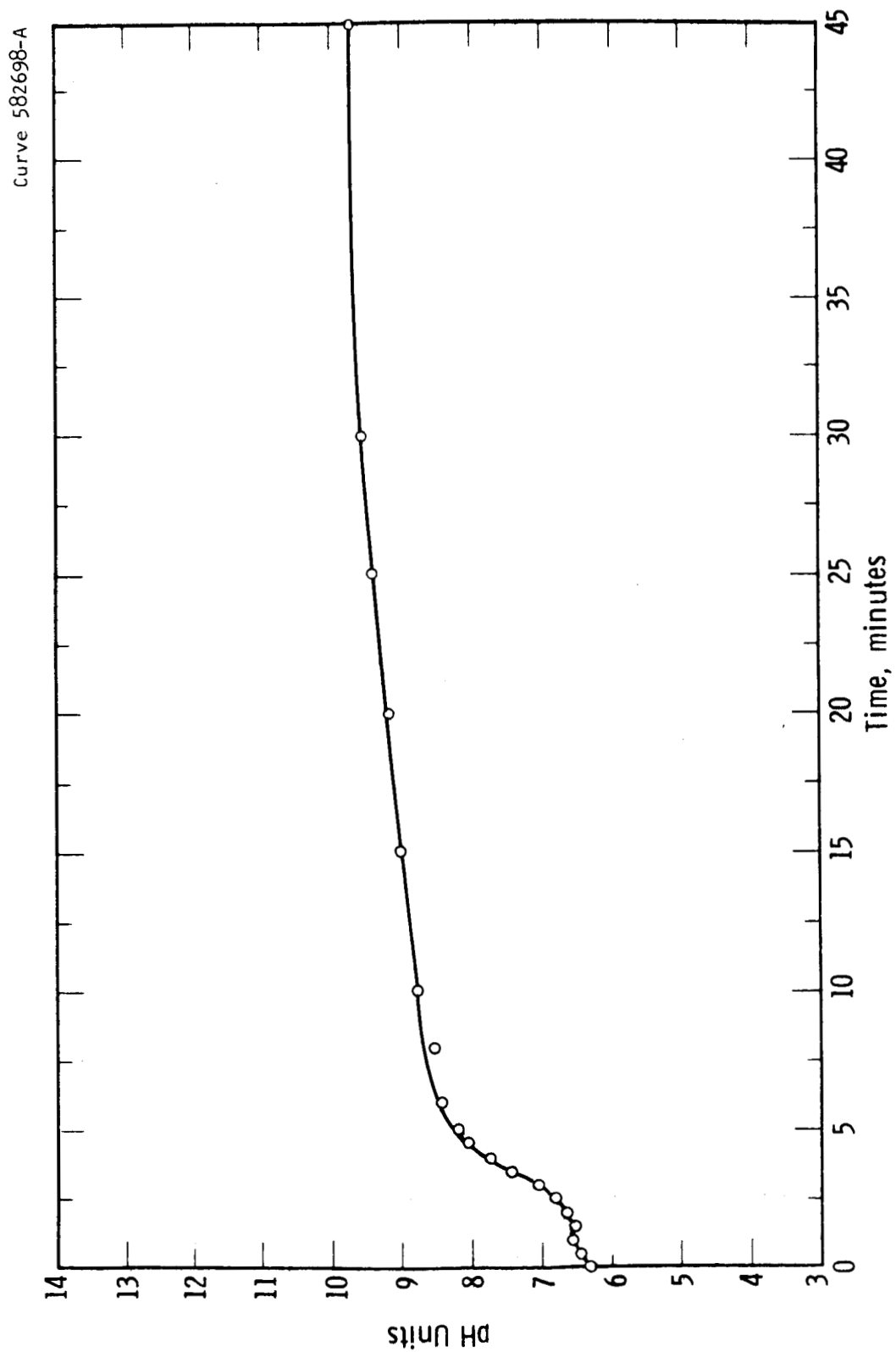


Fig. 11—Electrolyte diffusion time, 40% KOH, 26°C. Sample C58-4-3-5 extracted 6 hours in H₂O:
DMSO (25:75), ave. thickness 2.56 mils, Zeolon H : Polysulfone 20:15

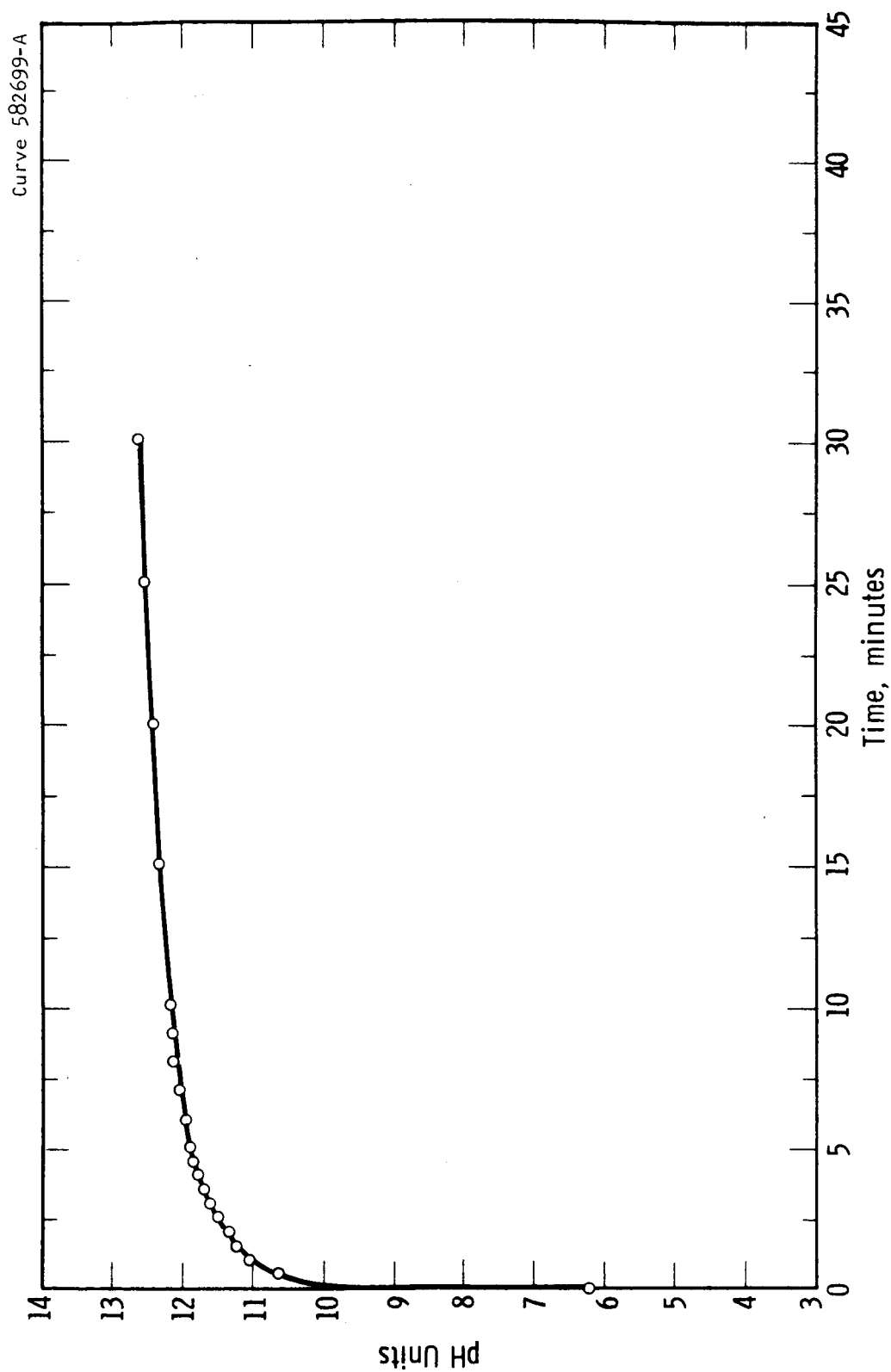


Fig. 12—Electrolyte diffusion time, 40% KOH, 26°C. Sample C58-4-3-8 extracted 1 min. in H_2O : dioxane (25:75), ave. thickness 2.34 mils, Zeolon H : Polysulfone 20:15

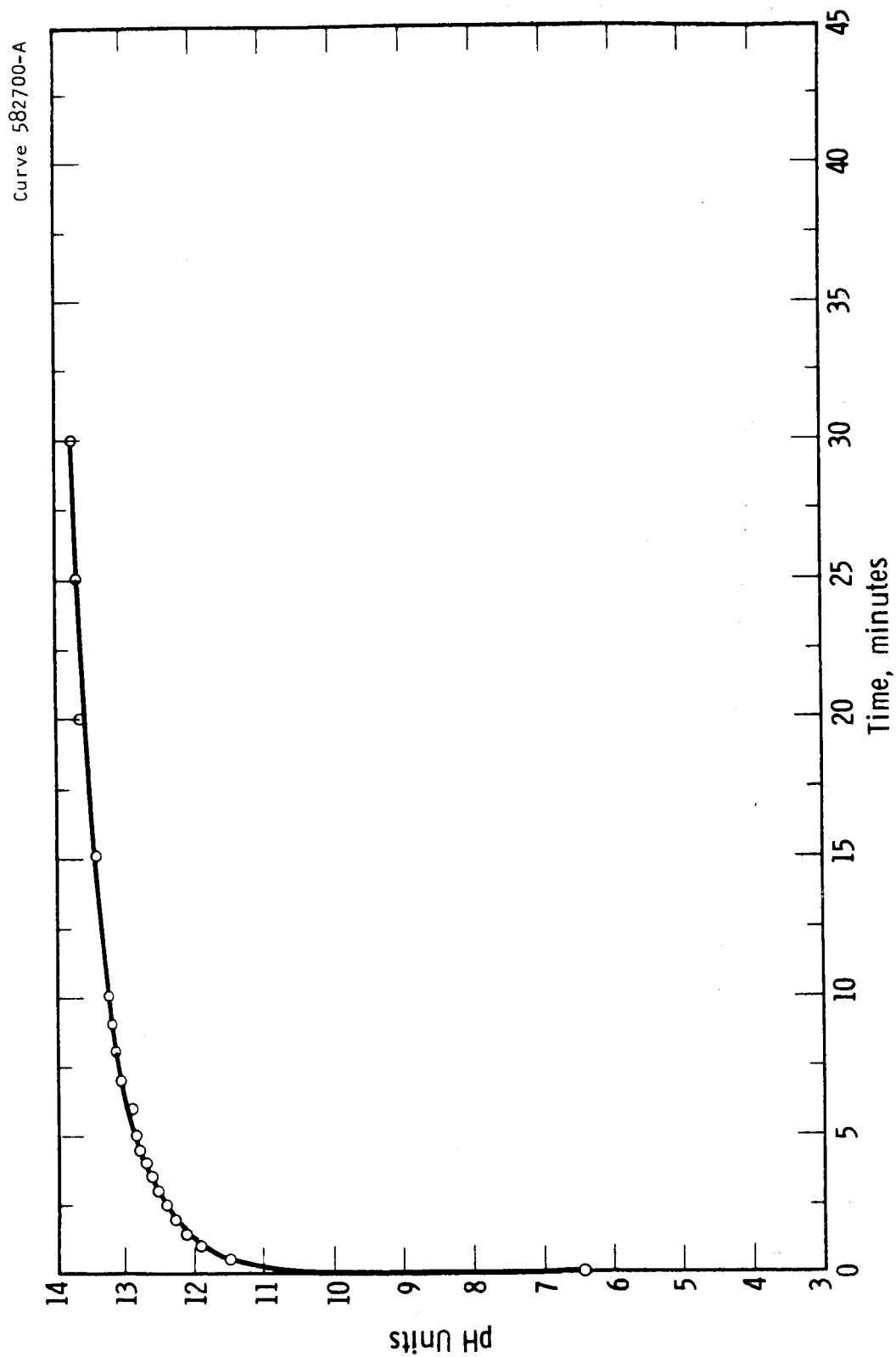


Fig. 13—Electrolyte diffusion time, 40% KOH, 26°C. Sample C58-4-3-8 extracted 30 min. in H₂O:
dioxane (25:75), ave. thickness 2.58 mils, Zeolon H : Polysulfone 20:15

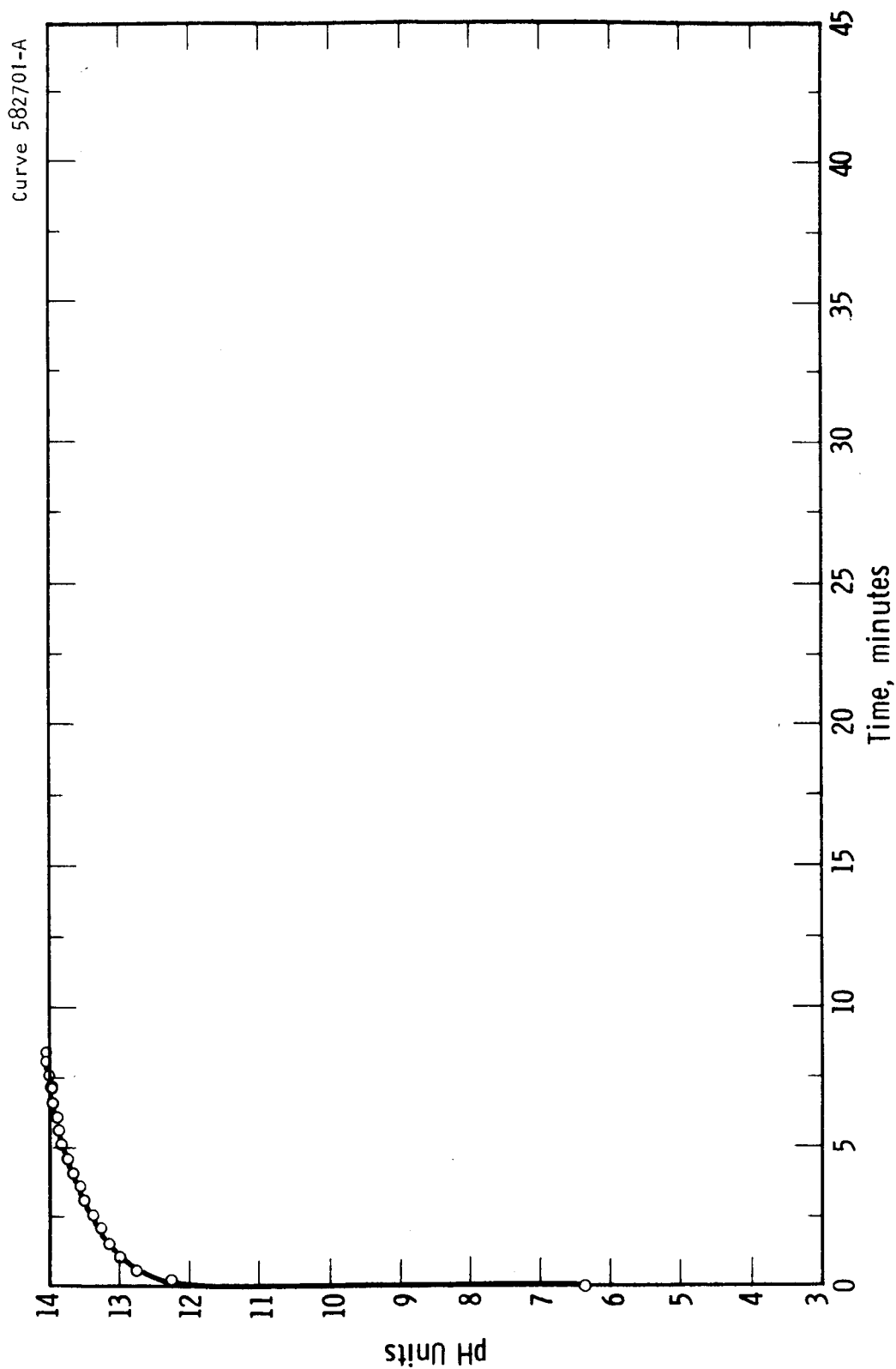


Fig. 14—Electrolyte diffusion time, 40% KOH, 26°C. Sample C58-4-3-8 extracted 2 hours in H_2O : dioxane (25:75), ave. thickness 2.72 mils, Zeolon H : Polysulfone 20:15

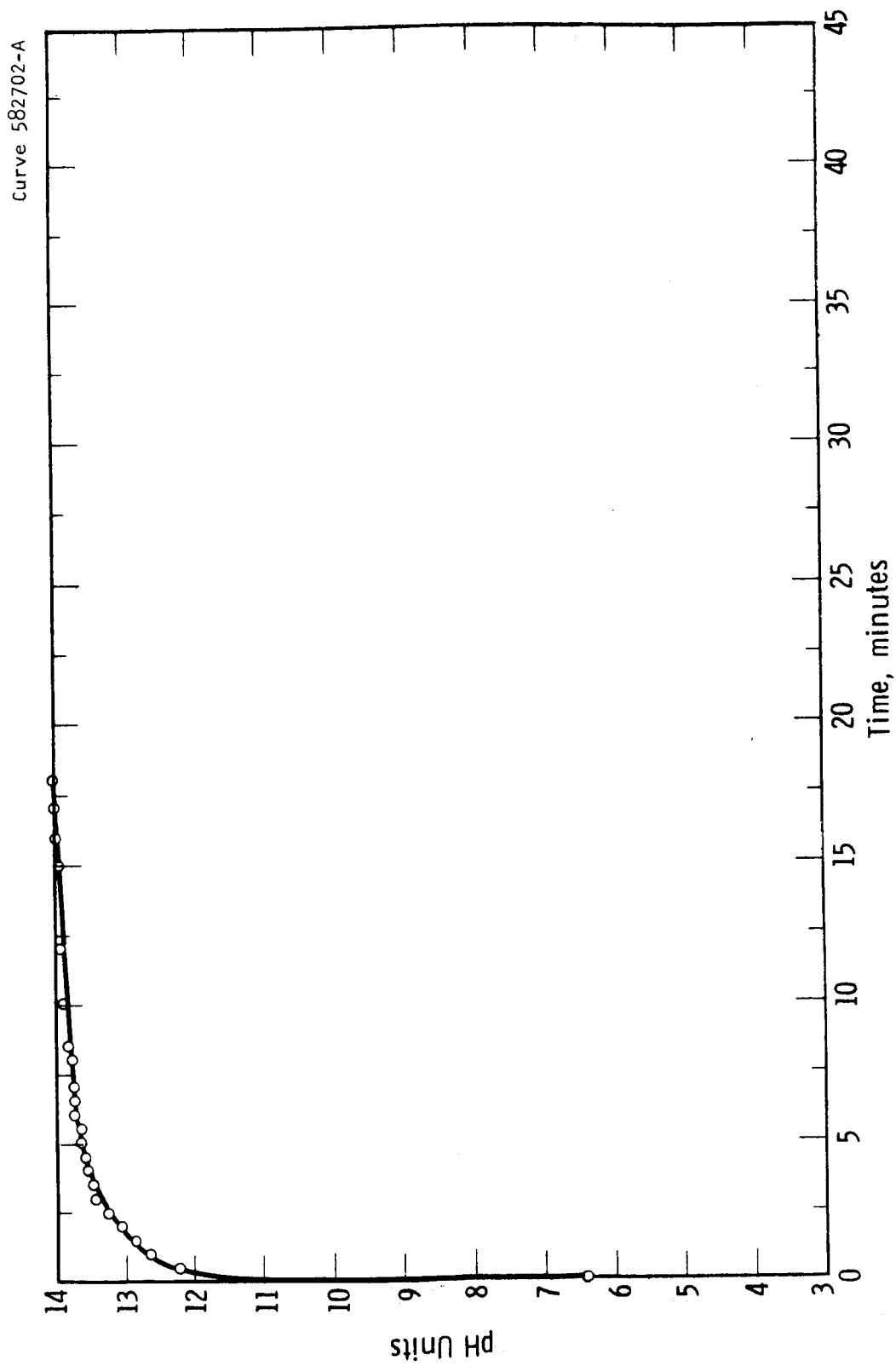


Fig. 15—Electrolyte diffusion time, 40% KOH, 26°C. Sample C58-4-3-8 extracted 6 hours in H_2O : dioxane (25:75), ave. thickness 3.02 mils, Zeolon H : Polysulfone 20:15

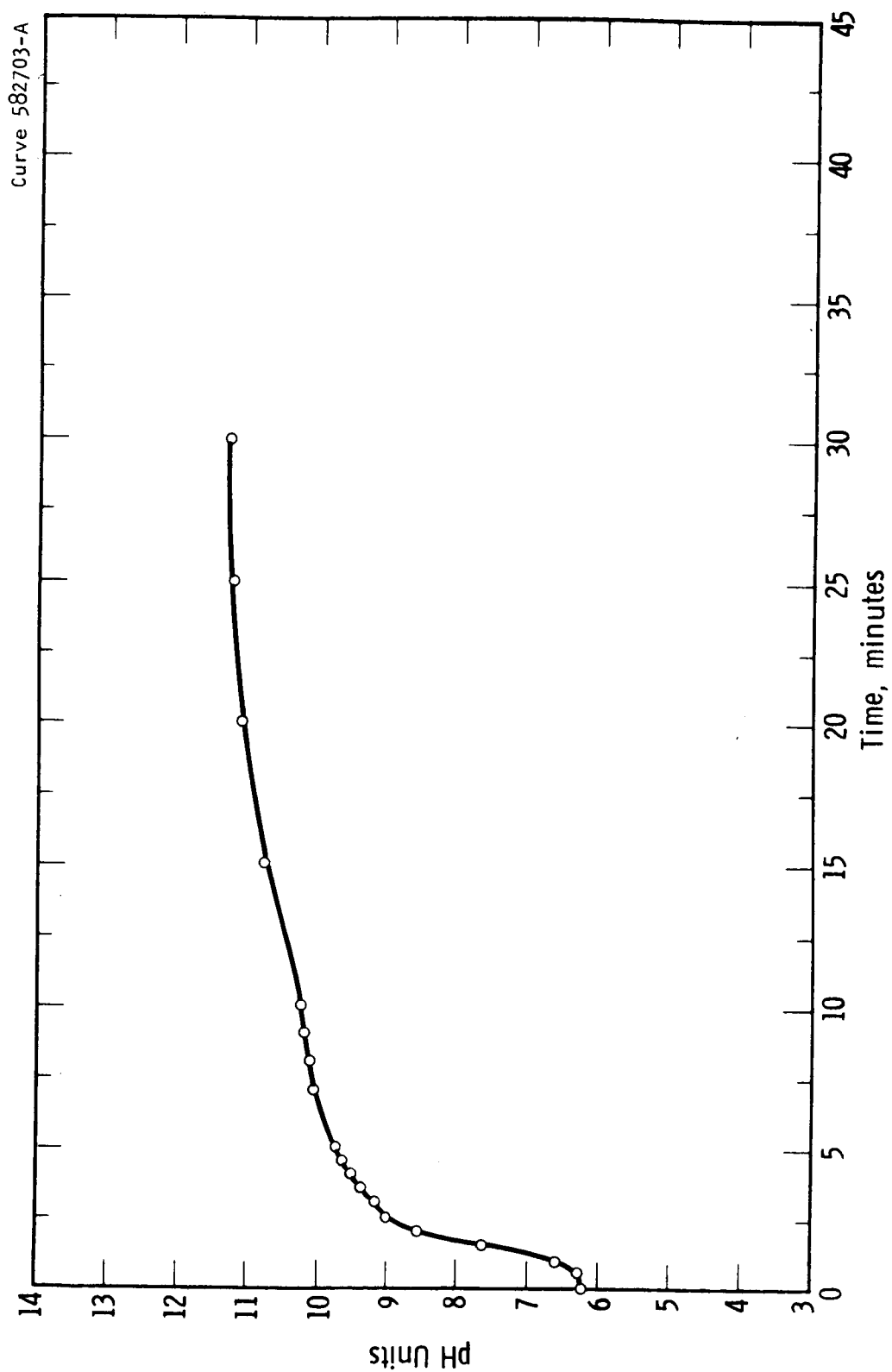


Fig. 16—Electrolyte diffusion time, 40% KOH, 26°C. Sample C58-4-3-9 extracted 1 min. in H₂O:
dioxane (75:25), ave. thickness 3.02 mils, Zeolon H : Polysulfone 20:15

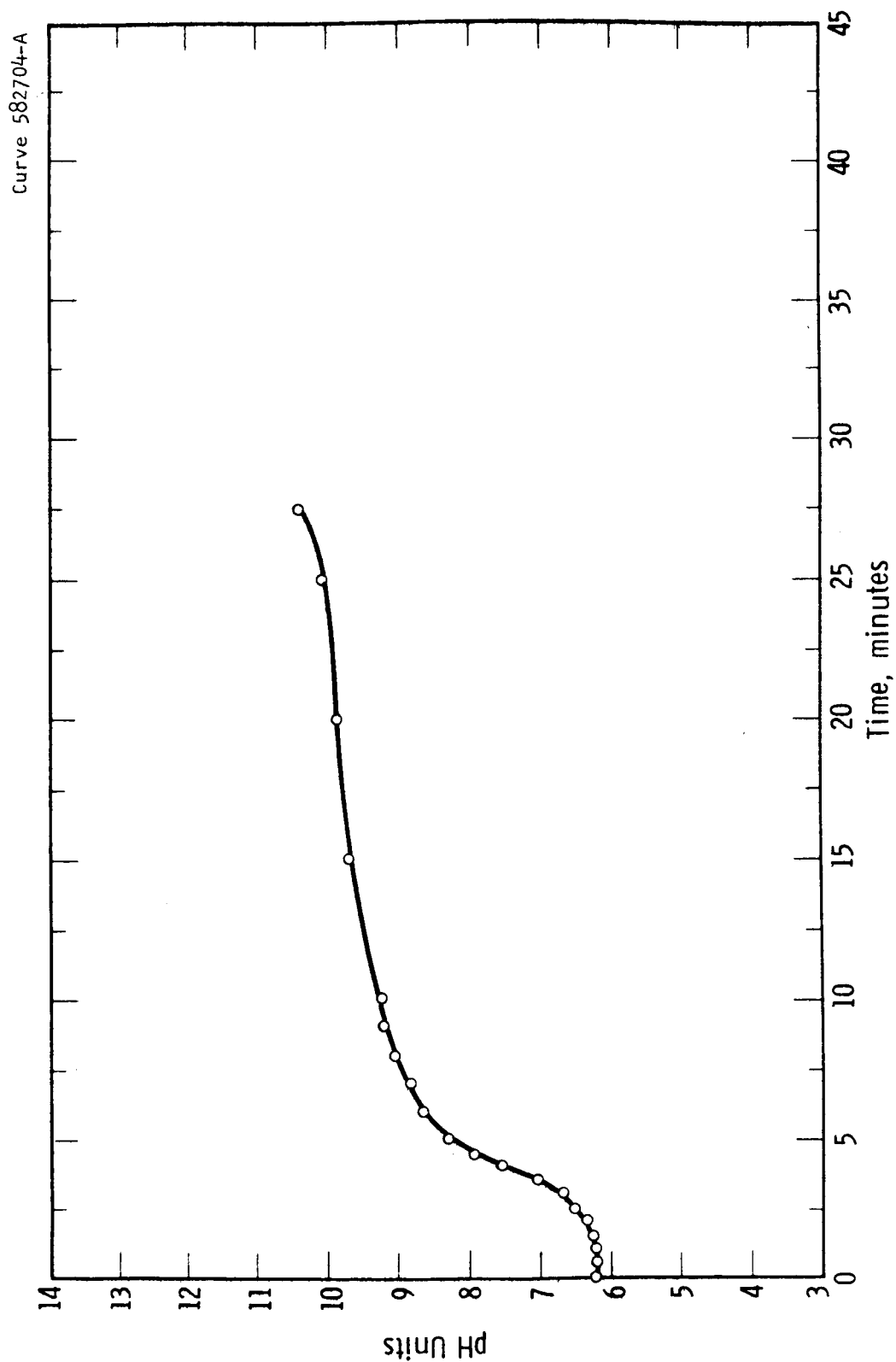


Fig. 17—Electrolyte diffusion time, 40% KOH, 26°C. Sample C58-4-3-9 extracted 30 min. in H₂O:
dioxane (75:25), ave. thickness 3.22 mils, Zeolon H : Polysulfone 20:15

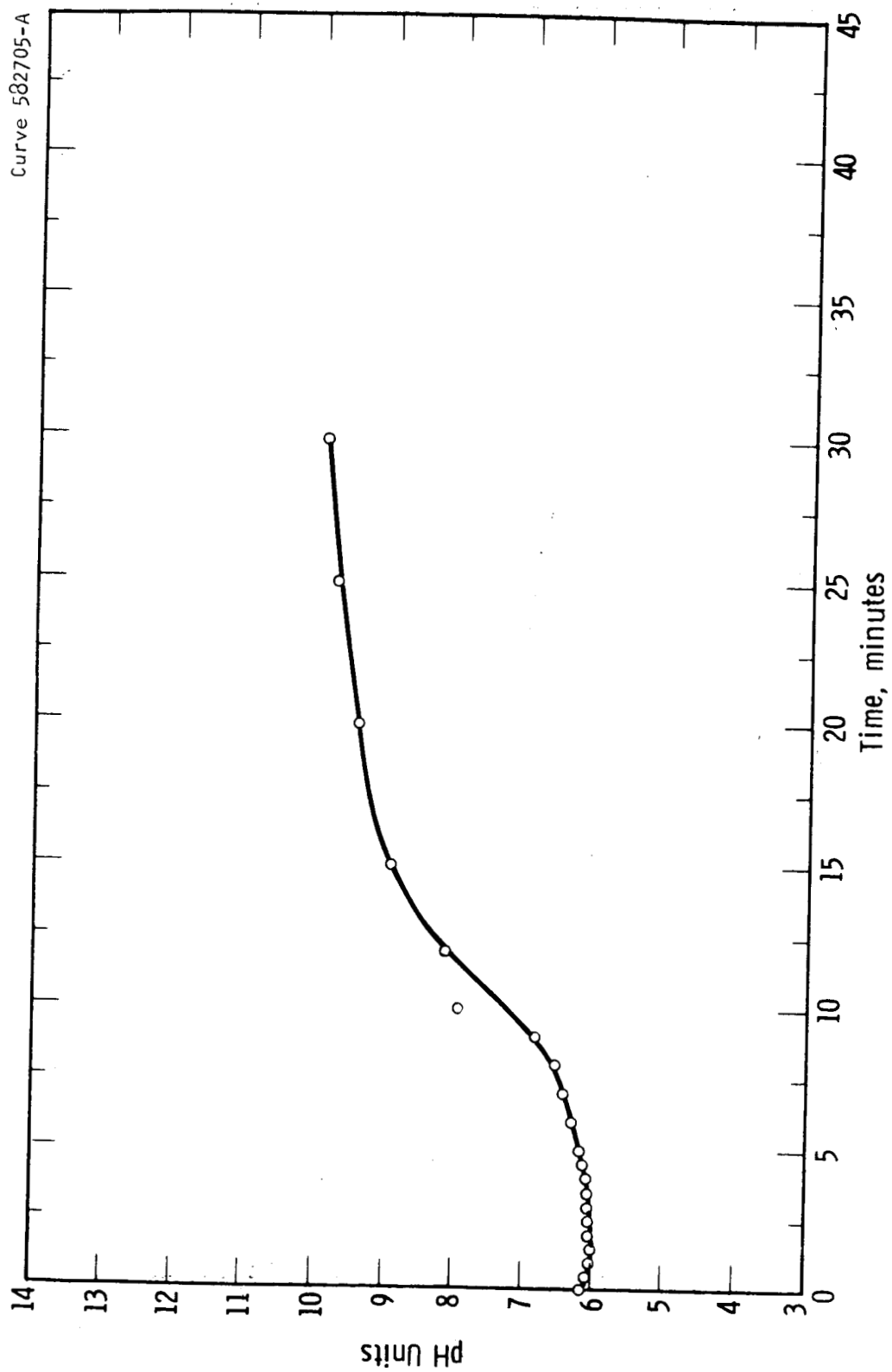


Fig. 18—Electrolyte diffusion time, 40% KOH, 26°C. Sample C58-4-3-9 extracted 2 hours in H₂O:
dioxane (75:25), ave. thickness 3.06 mils, Zeolon H : Polysulfone (20:15)

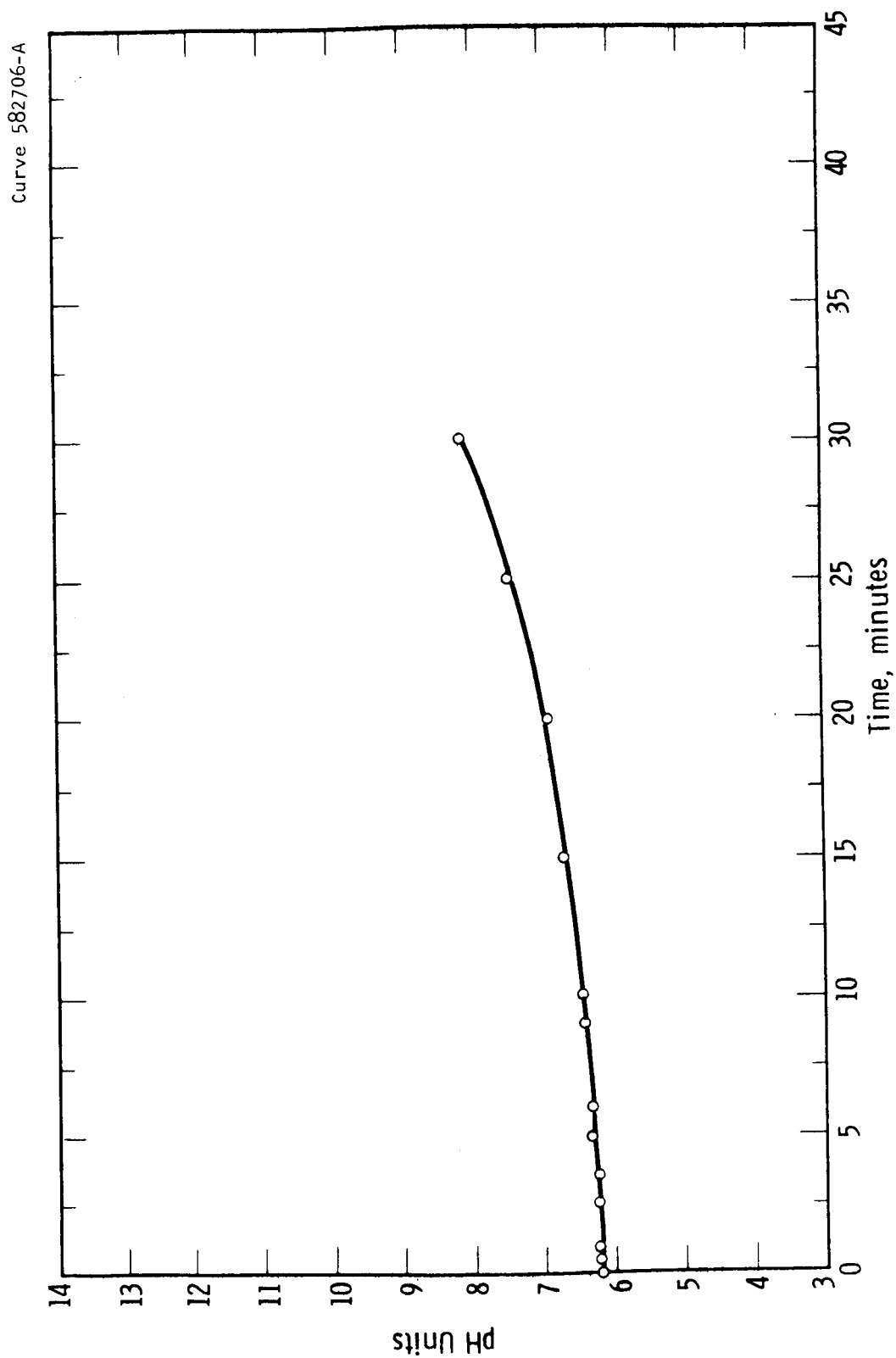


Fig. 19—Electrolyte diffusion time, 40% KOH, 26°C. Sample C58-4-3-9 extracted 6 hours in H₂O: dioxane (75:25), ave. thickness 3.08 mils, Zeolon H : Polysulfone 20:15

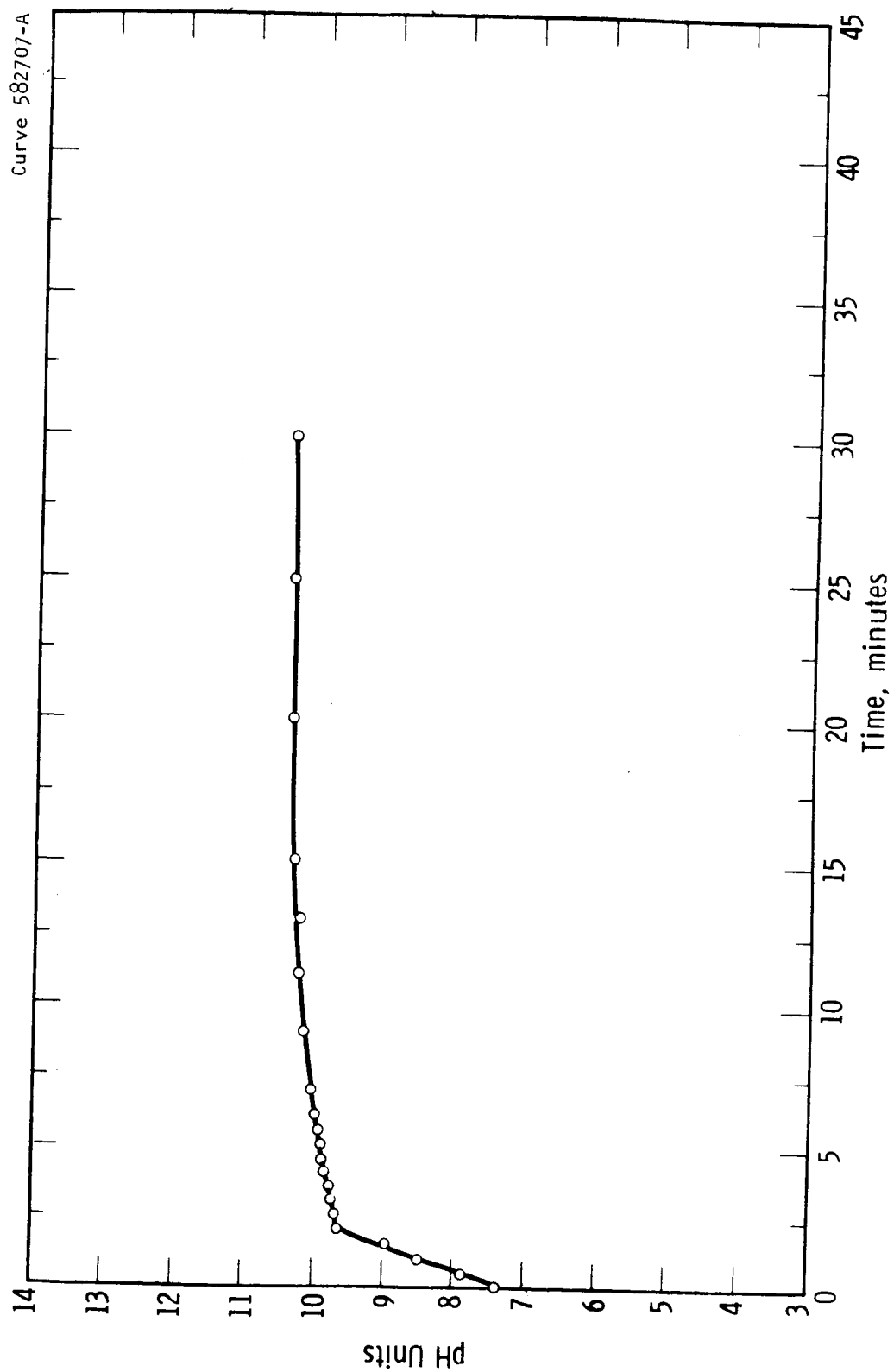


Fig. 20—Electrolyte diffusion time, 40% KOH, 26°C. Sample C58-4-3-12, extracted 1 min. in H₂O:
acetic acid (75:25), ave. thickness 3.08 mils, Zeolon H : Polysulfone 20:15

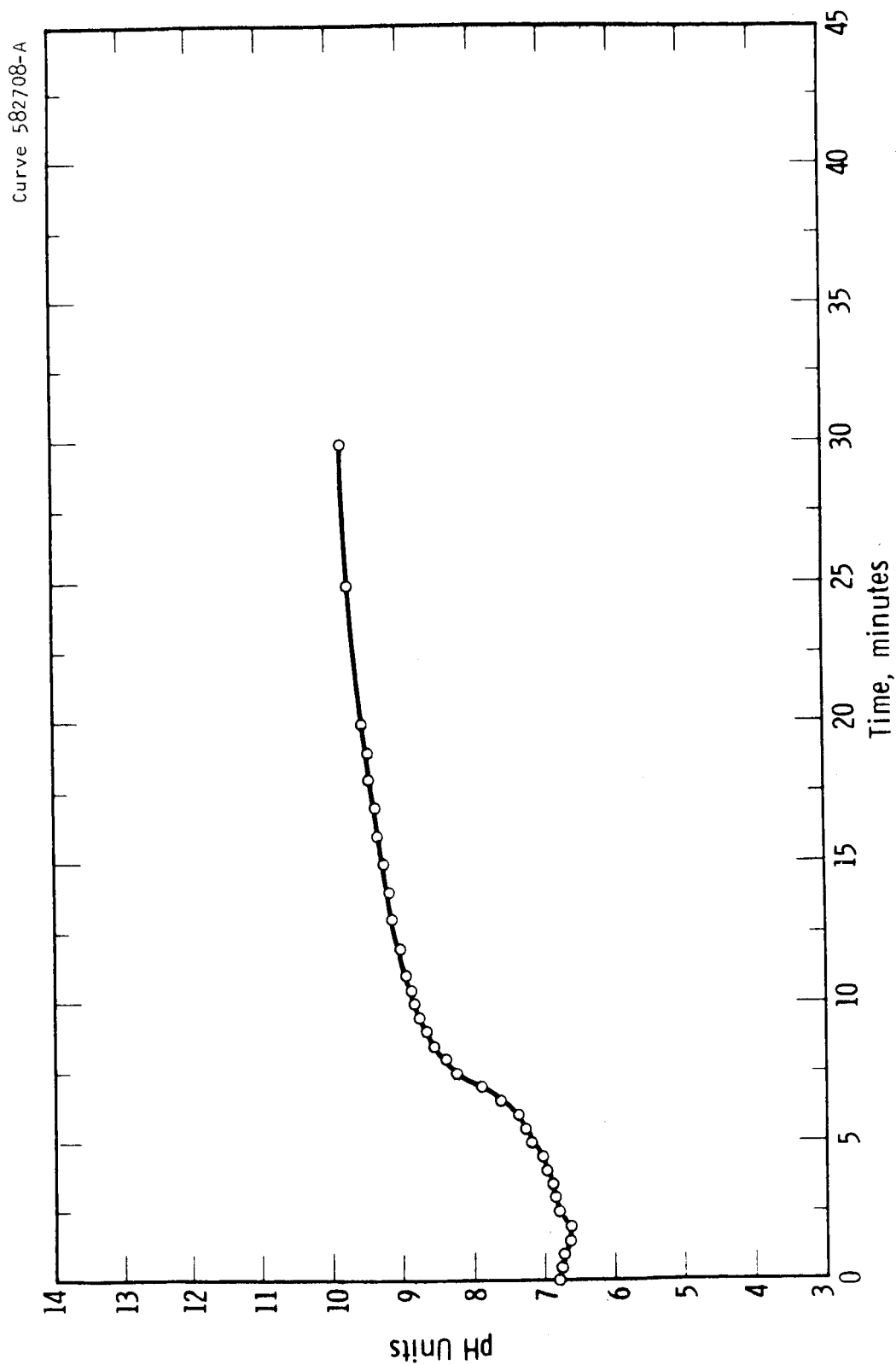


Fig. 21—Electrolyte diffusion time, 40% KOH, 26°C. Sample C58-4-3-12 extracted 30 min. in H_2O : acetic acid (75:25), ave. thickness 3.7 mils, Zeolon H : Polysulfone 20:15

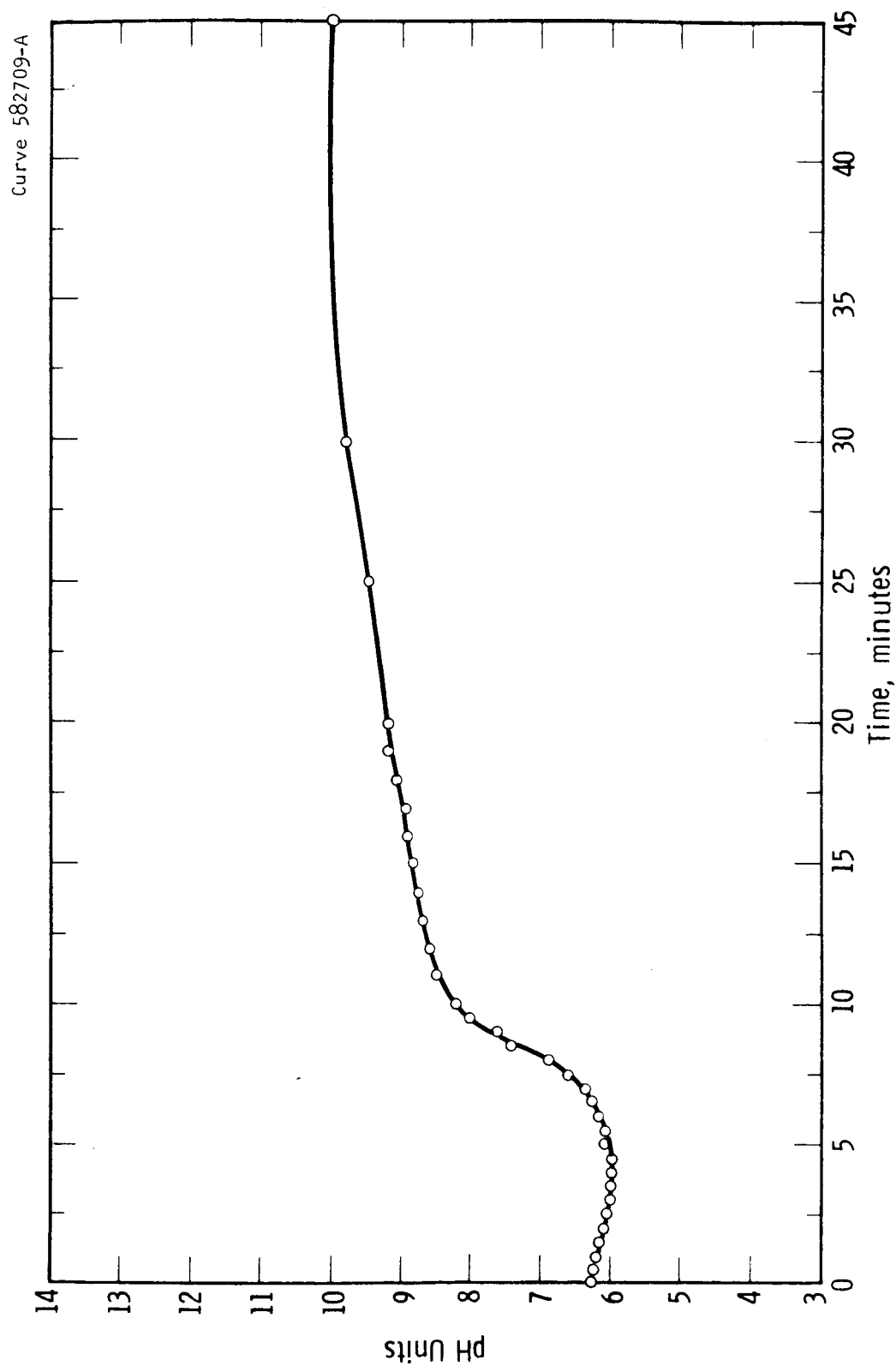


Fig. 22—Electrolyte diffusion time, 40% KOH, 26°C. Sample C58-4-3-12, extracted 2 hours in H₂O:
acetic acid (75:25), ave. thickness 2.9 mils, Zeolon H : Polysulfone 20:15

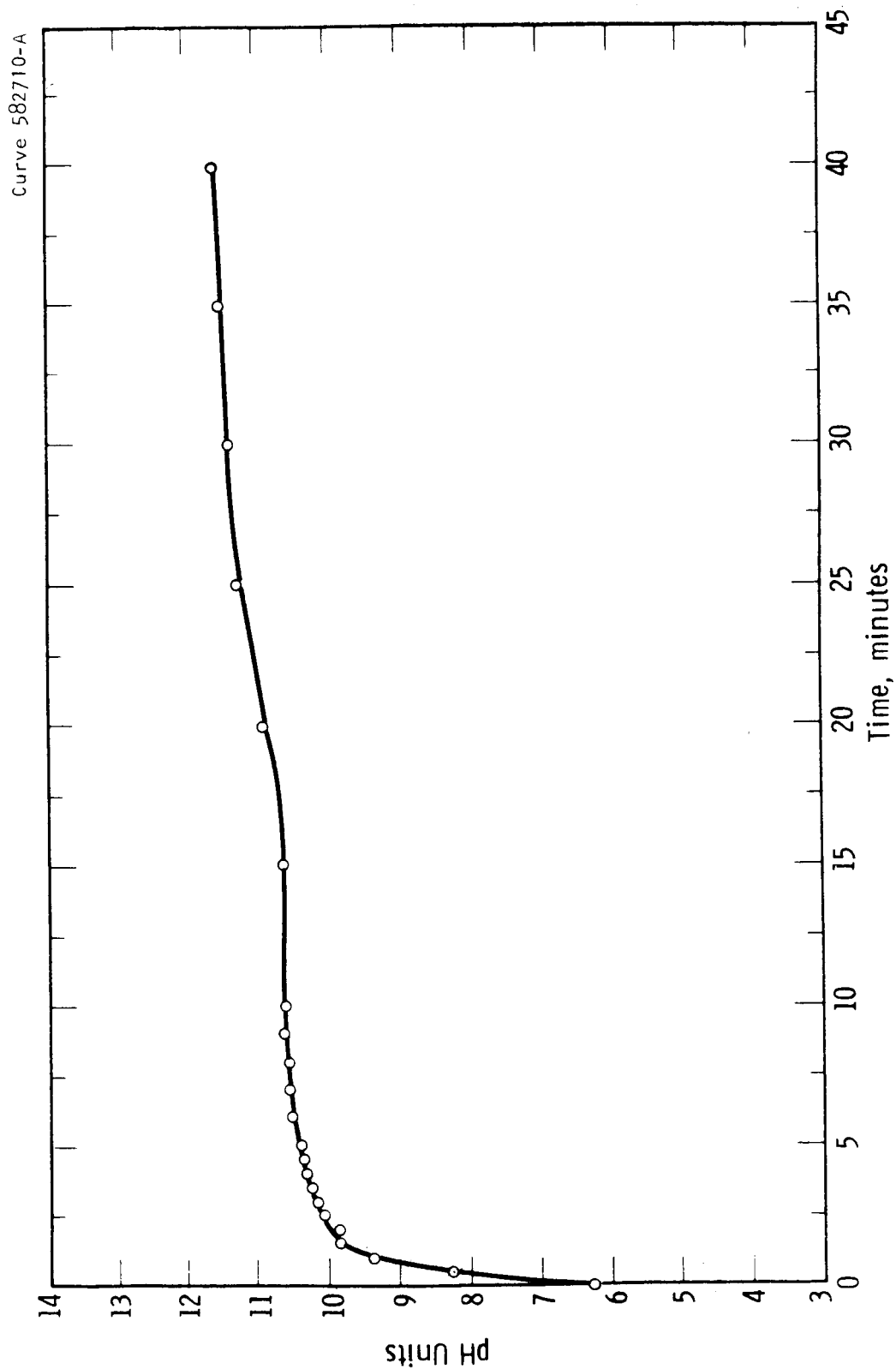


Fig. 23—Electrolyte diffusion time, 40% KOH, 26°C. Sample C58-4-3-12, extracted 6 hours in H₂O:
acetic acid (75:25), ave. thickness 3.4 mils, Zeolon H : Polysulfone 20:15

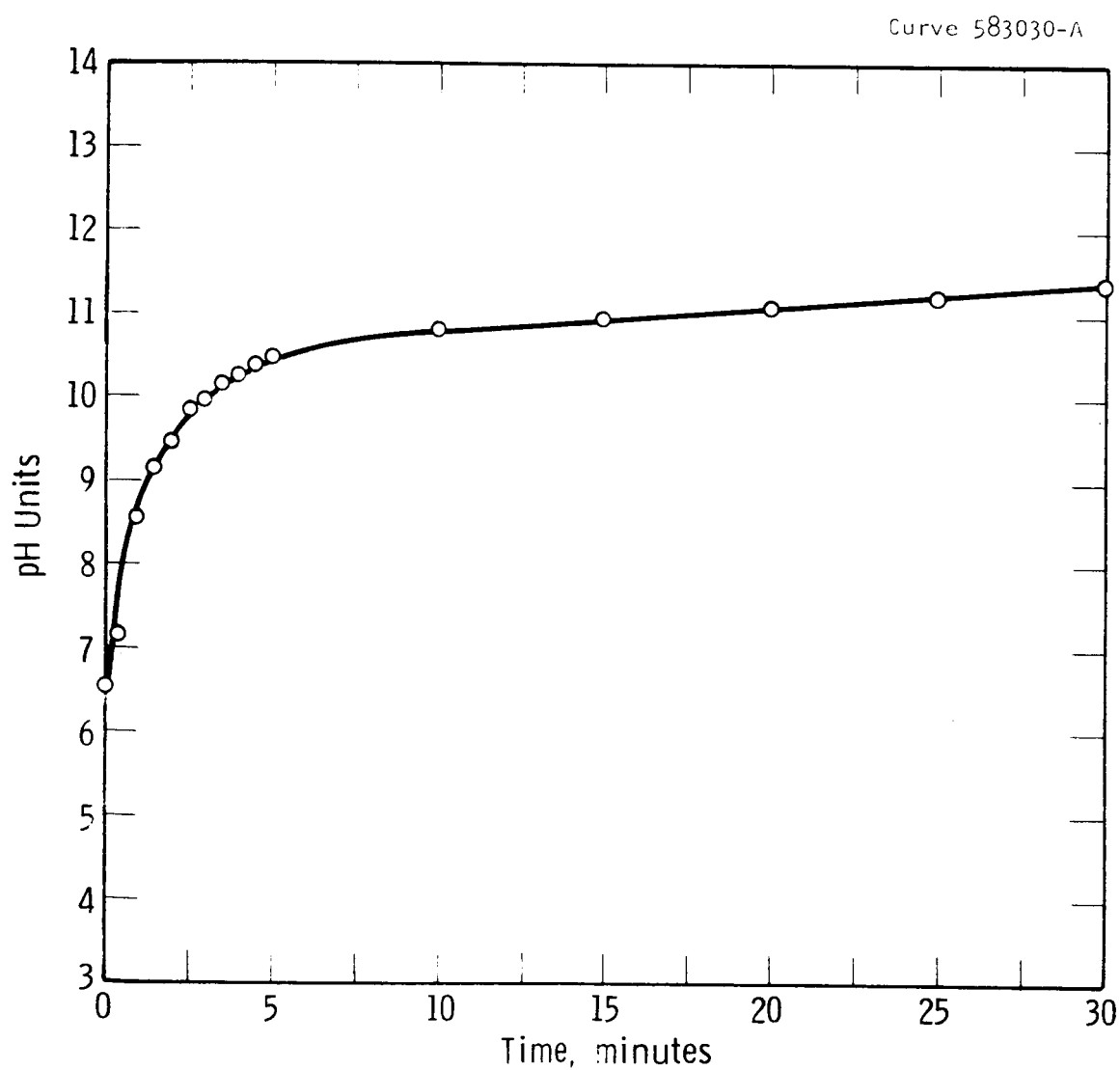


Fig. 24—Electrolyte diffusion time, 40% KOH, 26°C. Air-dried sample.
Ave. thickness: 2.7 mils, BioRad zirconia (20-50 mesh):
PS ratio = 20:15

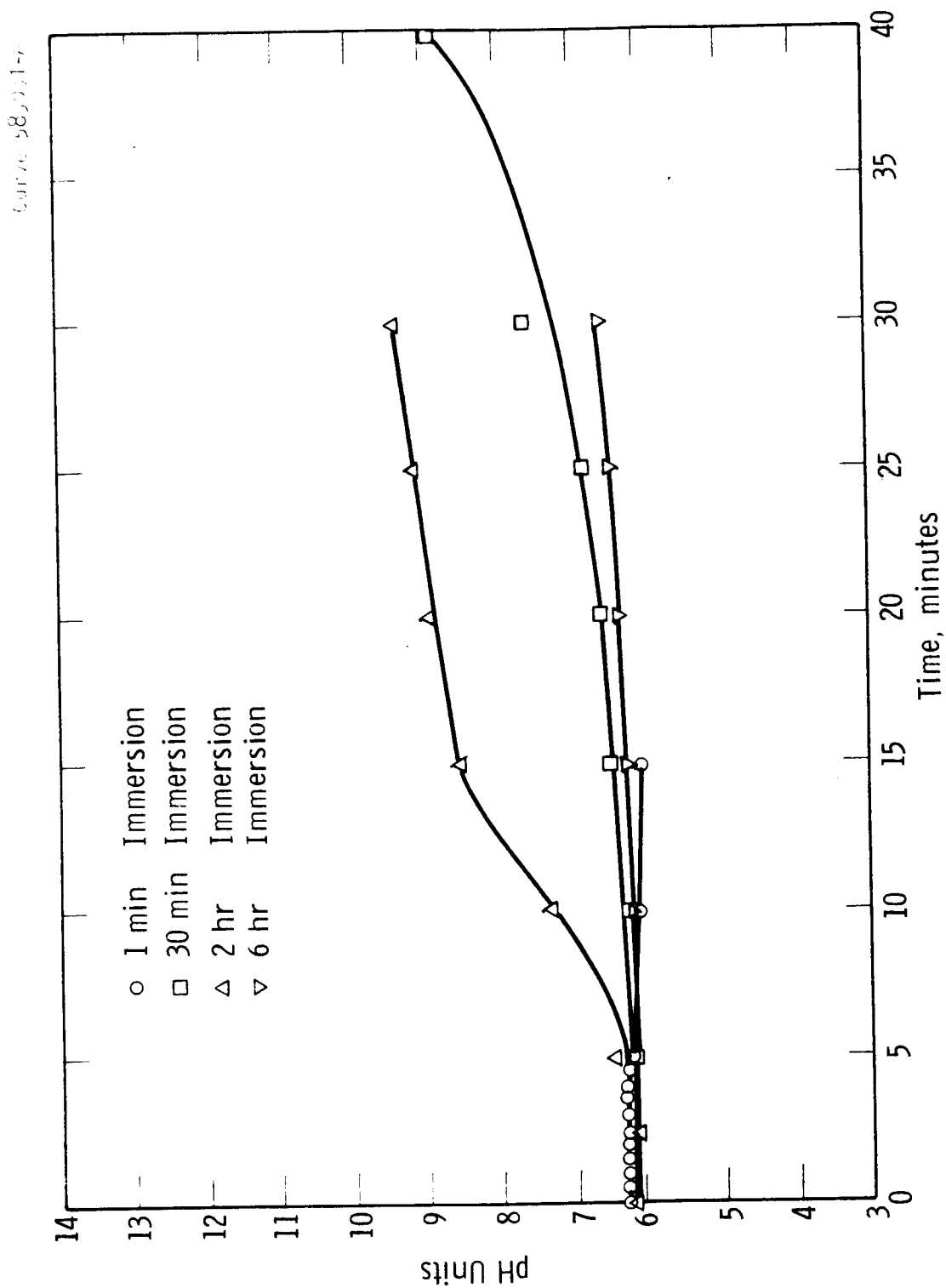


Fig. 25—Electrolyte diffusion time, 40% KOH, 26°C. Series C58-12-2-1, extracted for 1 min, 30 min, 2 hours and 6 hours in 50:50 H₂O:DMAC. Ave. thickness: 3 mils

BioRad zirconia (20-50 mesh) : PS ratio = 20:15

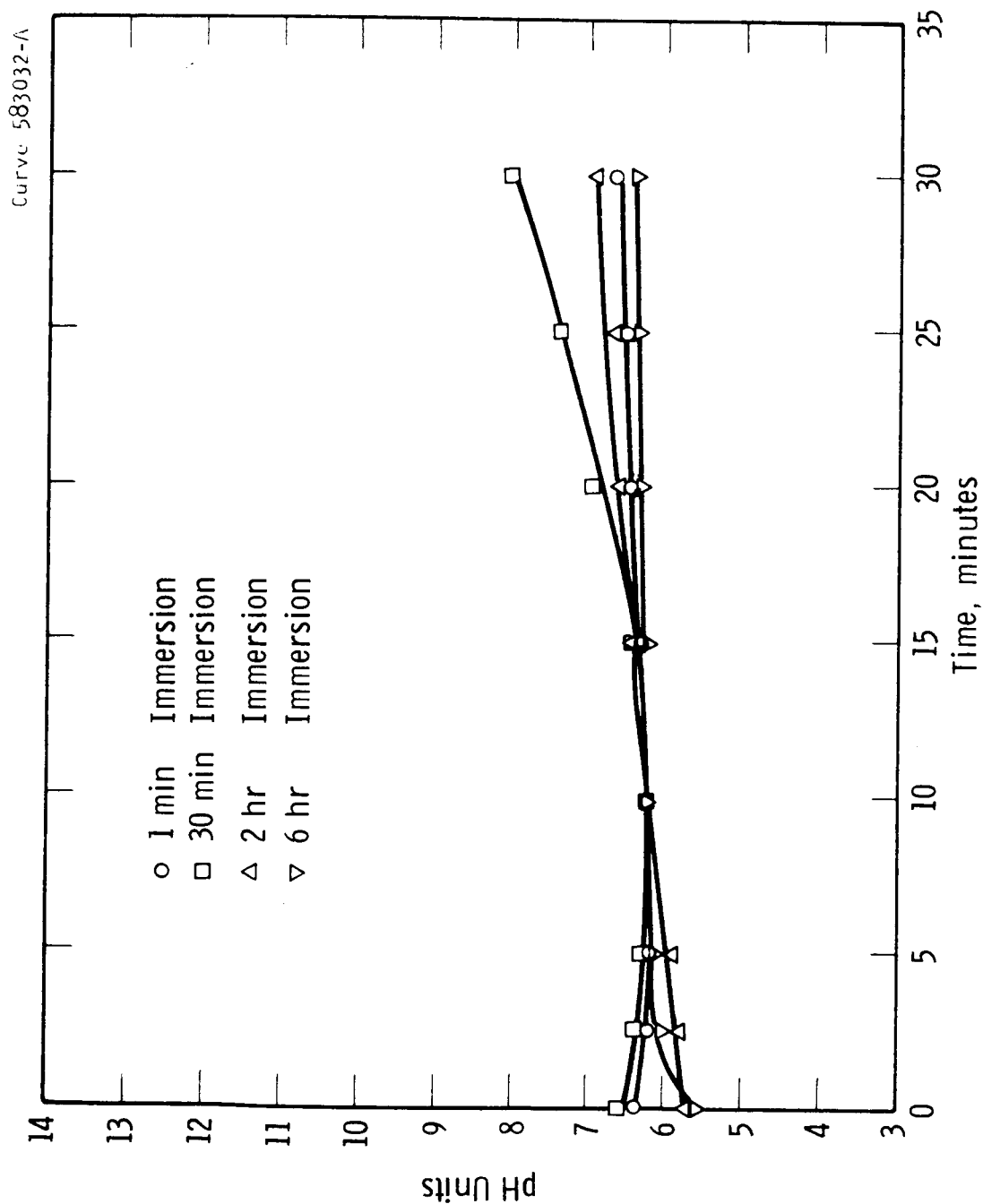


Fig. 26—Electrolyte diffusion time, 40% KOH, 26°C. Series C58-12-2-2, extracted for 1 min, 30 min, 2 hours and 6 hours in 25 : 75 H₂O : DMAC.

Ave. thickness: 2.9 mils, BioRad zirconia (20 : 50 mesh) : PS ratio = 20 : 15

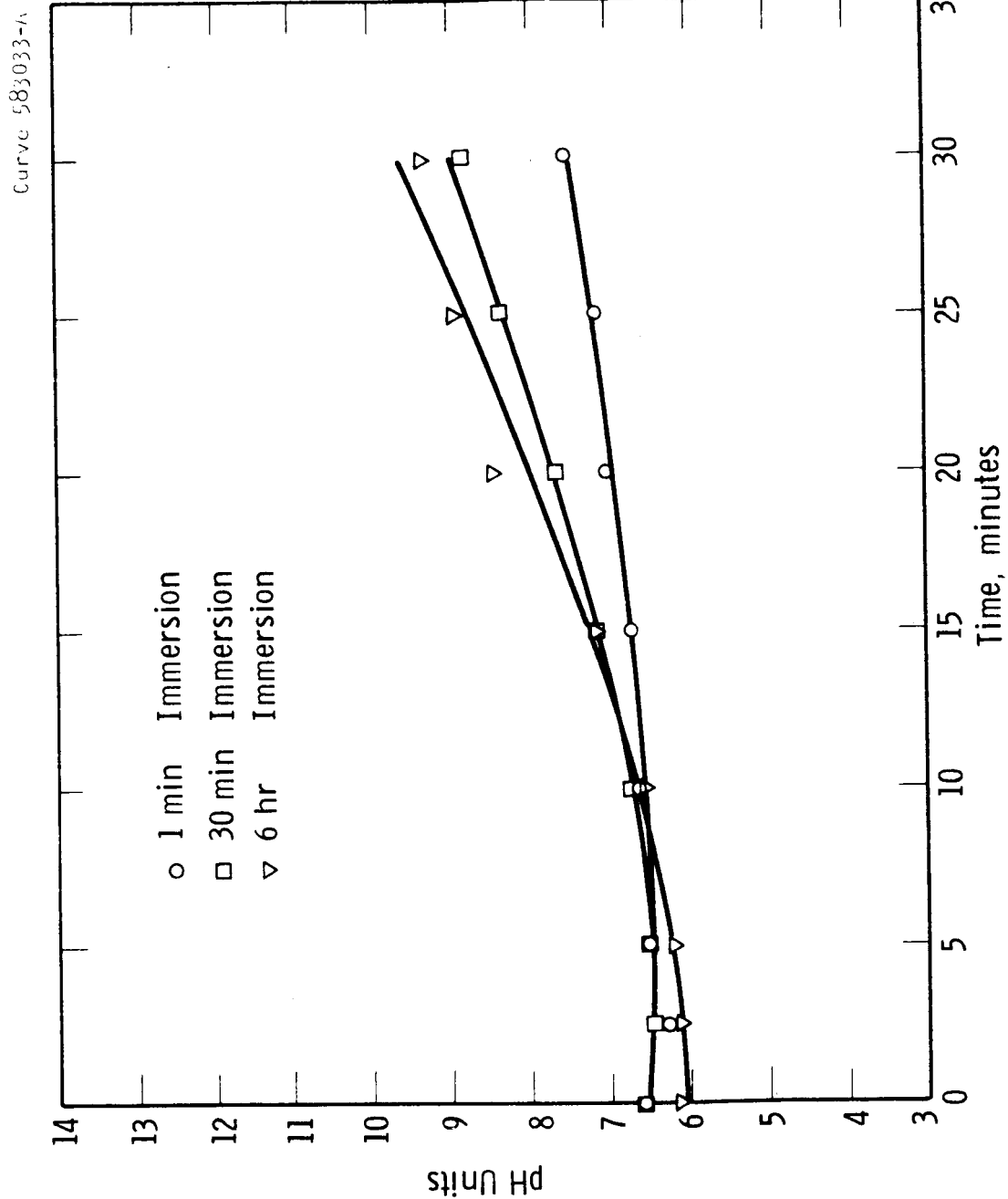


Fig. 27—Electrolyte diffusion time, 40% KOH, 26°C. Series C58-12-2-3, extracted for 1 min, 30 min and 6 hours in 75:25 H₂O:DMAC. Ave. thickness: 3.1 mils

BioRad zirconia (20-50 mesh): PS ratio = 20:15

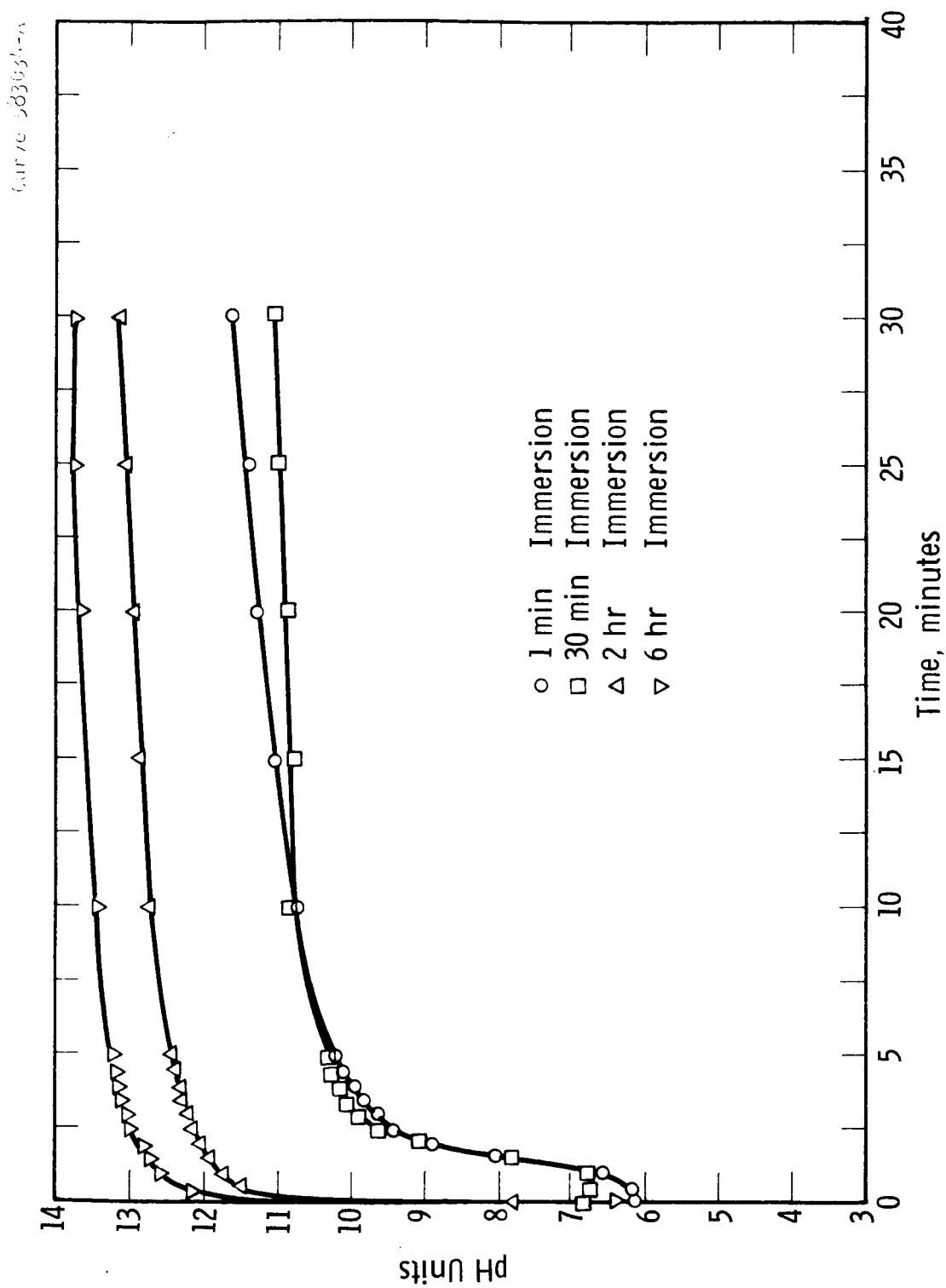


Fig. 28—Electrolyte diffusion time, 40% KOH, 26°C. Series C58-12-2-8, extracted for 1 min, 30 min, 2 hours and 6 hours in 27 : 75 H₂O : dioxane. Ave. thickness: 3 mils

BioRad zirconia (20-50 mesh): PS ratio = 20 : 15

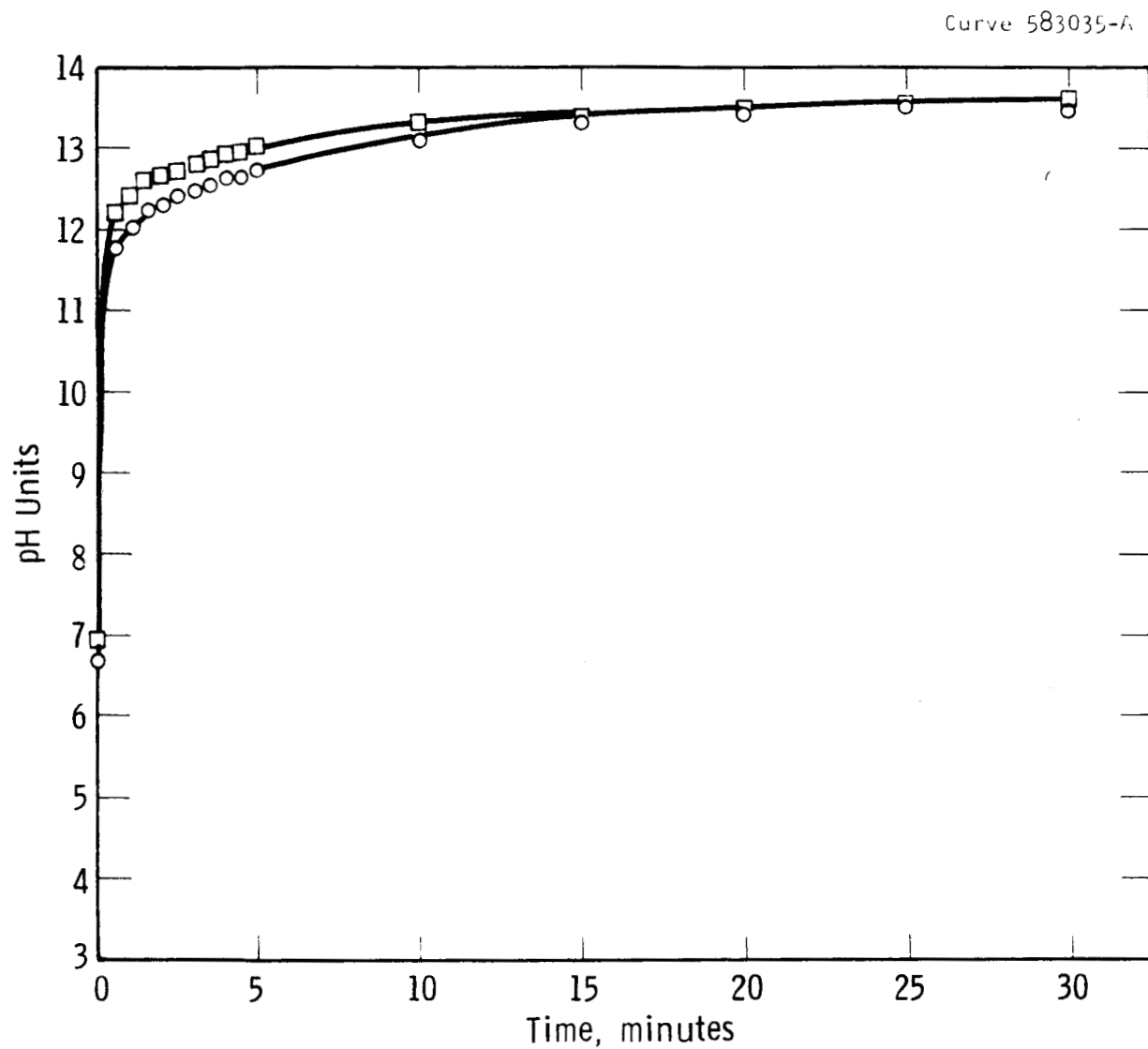


Fig. 29—Electrolyte diffusion time, 40% KOH, 26°C. Continuous run C58-14-1, 3 mil gap on gardner knife two spots measured. Dried TAM zirconia: PS ratio = 20 : 15, extractant = 1 : 2 H₂O : dioxane

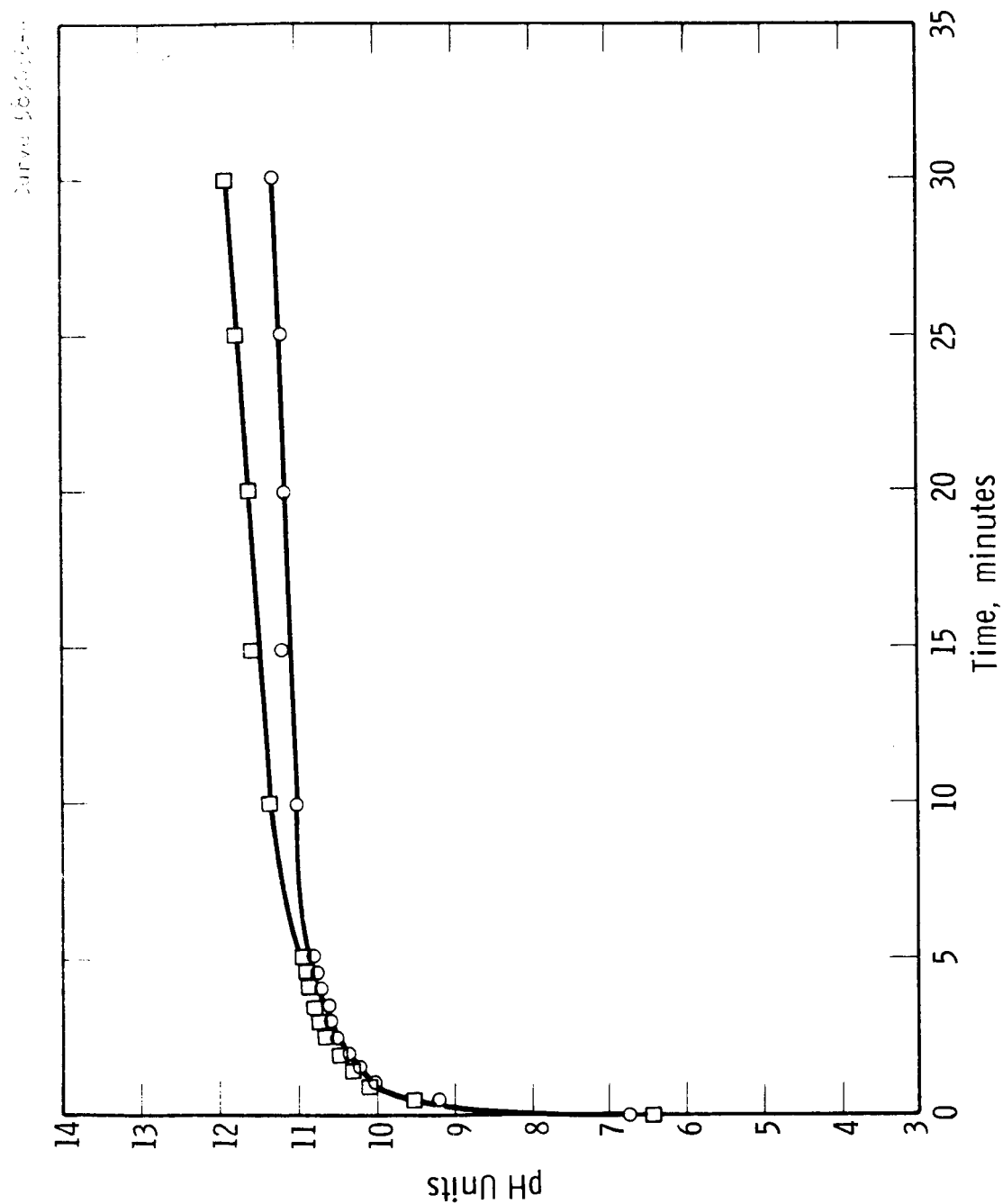


Fig. 30—Electrolyte diffusion time, 40% KOH, 26°C. Continuous run C58-14-1, 4 mil gap on gardner knife, double coating, two spots measured. Dried TAM zirconia: PS ratio = 20:15, extractant = 1:2 H₂O : dioxane

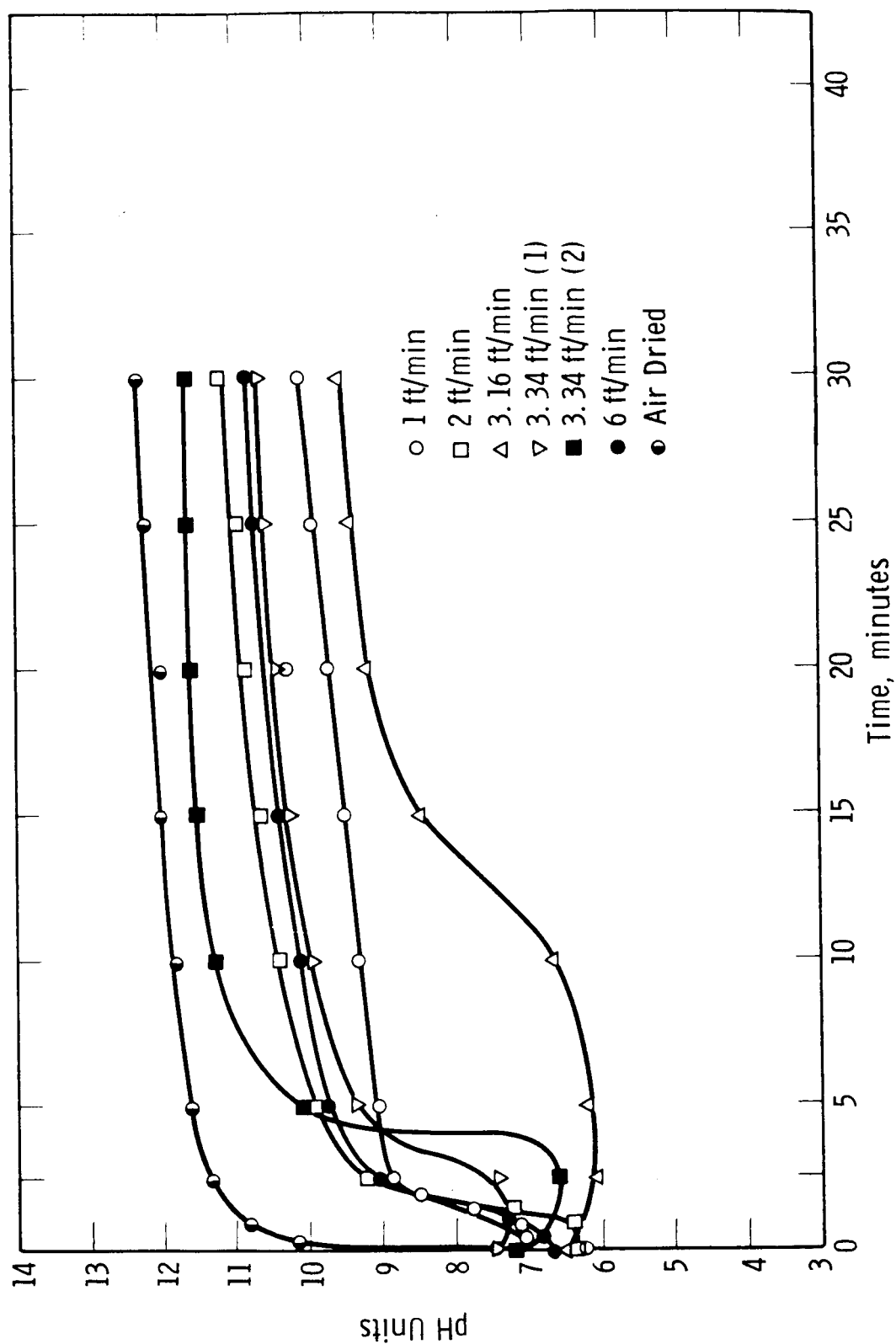


Fig. 31—Electrolyte diffusion time, 40% KOH. Continuous run C58-18-1, 4 mils gap at knife.
BioRad zirconia: PS ratio = 30:15, extractant = 1:3 H₂O:dioxane

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Security Classification

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1 ORIGINATING ACTIVITY (Corporate author) Westinghouse Electric Corporation Beulah Road, Churchill Boro Pittsburgh, Pennsylvania 15235		2a REPORT SECURITY CLASSIFICATION	
		2b GROUP	
3 REPORT TITLE SEPARATOR DEVELOPMENT FOR A HEAT STERILIZABLE BATTERY			
4 DESCRIPTIVE NOTES (Type of report and inclusive dates) Fifth Quarterly Report, July 1 to September 30, 1967			
5 AUTHOR(S) (Last name, first name, initial) Scala, L. C.			
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13. ABSTRACT A continuous coating apparatus to prepare sterilizable battery separators has been built, tested and put into the production of several feet of separator material which exhibited satisfactory properties. The effect of time of immersion in extractant solutioning upon separator composites has been investigated using formulations in which the filler was either Zeolon H or coarse Zirconia. The best extracting solutions contained dioxane. Generally no correlation was found between some separator properties and time of immersion.			

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14 KEY WORDS	LINK A		LINK B		LINK C	
	ROLE	WT	ROLE	WT	ROLE	WT
Battery, separators, sterilization, continuous, coating, fabrication, extraction, polysulfone, polypropylene, tape, testing, dioxane, resistivity, ion, diffusion, zirconia						

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